IDAHO BLM

TECHNICAL BULLETIN

PILOT SAMPLING FOR HEAVY METALS IN FISH FLESH FROM KILLARNEY LAKE, COEUR d'ALENE RIVER SYSTEM, IDAHO

Prepared for

COEUR d'ALENE DISTRICT
BUREAU OF LAND MANAGEMENT

by

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Technical Bulletin 90-6

September 1990

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ACKNOWLEDGMENTS

We thank the U. S. Bureau of Land Management for funding this project. We thank Bruce Rich for his assistance in collecting the fishes in Killarney Lake, Kelly Florance for sample preparation, and Bill Warner in the Veterinary Science Department, at the University of Idaho, Moscow, for conducting the metal analyses.

ABSTRACT

Mining and smelting activities on the South Fork of the Coeur d' Alene River in northern Idaho since 1885 have contributed massive amounts of metals-rich tailings to the river. Recent environmental studies have generally demonstrated an improved condition in the Coeur d'Alene River, lateral lakes, and downstream Lake Coeur d' Alene since the early monitoring. We sampled largemouth bass (*Micropterus salmoides*), northern pike (*Esox lucius*), black crappie (*Pomoxis nigromaculatus*), brown bullhead (*Ictalurus nebulosus*), and yellow perch (*Perca flavescens*) from Killarney Lake, a lateral lake on the Coeur d' Alene River in northern Idaho and analyzed edible flesh for concentrations of zinc, lead, mercury, cadmium, copper, and arsenic. Mean concentrations (n=10) of zinc were highest in yellow perch (4.79 mg/kg wet weight) and lowest in brown bullheads (2.95 mg/kg), considerably lower than the 45 mg/kg action level from the literature. Mean concentrations (n=10) of copper ranged from 0.265 mg/kg in black crappie to 0.31 mg/kg in yellow perch, northern pike and largemouth bass, also well below the recommended action level of 20 mg/kg. Mean concentrations (n=10) of lead ranged from 0.014 mg/kg in largemouth bass to 0.183 mg/kg in brown bullheads, compared to the 3.50 mg/kg action level. Mean concentrations (n=10) of cadmium ranged from 0.018 to 0.04 mg/kg, highest in brown bullheads and lowest in northern pike. Action levels listed in the literature for cadmium were 0.07 mg/kg. Mean mercury concentrations ranged from 0.034 mg/kg in brown bullheads to 0.093 mg/kg in yellow perch, also less than the 1 mg/kg action level. Concentrations of arsenic were generally below detection limits (<0.04

mg/kg) for all species but varied considerably between two laboratories which conducted analysis on replicate tissue samples. Comparisons of all species of fishes with the metals tested resulted in significant increases in body content as a function of size in largemouth bass for copper and mercury in yellow perch and zinc in black crappie. Comparisons of fish flesh content with allowable human intakes resulted in estimation of a maximum allowable consumption of 3.1 kg/week of fish from Killarney Lake. Tolerance limits calculated for the various flesh concentrations of metals indicated that we were 99% confident that 99% of the populations were at or below action levels for the species sampled for zinc, copper, and lead. Tolerance limits for cadmium in yellow perch and brown bullheads exceeded the recommended action limits, while those in northern pike and black crappie were at the action level. Based on our sample results from 10 fish, additional sampling is needed to adequately describe the distribution of lead in brown bullheads (N = 50) and yellow perch (N = 26) from Killarney Lake. Differences in detection limits of heavy metals, differences in concentrations from the same fillet from two laboratories, and between-site variation all suggest a cautious approach to the interpretation of heavy metal analysis data from surface waters.

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INTRODUCTION

Mining and smelting activities on the South Fork of the Coeur d' Alene River in northern Idaho since 1885 have contributed massive amounts of tailings to the river. Prior to construction of settling ponds, the river transported high loads of dissolved and suspended metals and sediment with deposition in the downstream floodplain and lateral lakes system. The U.S. Environmental Protection Agency (EPA) has been monitoring water quality in the river since passage of the Clean Water Act in 1972. These studies have generally indicated an improved condition in the river, lateral lakes, and downstream Lake Coeur d' Alene since the early 1970's (Hornig et al. 1988).

Heavy metal concentrations in fishes from the Coeur d' Alene River system are of concern because of the importance of sports fishing in the lateral lakes. With construction of Post Falls Dam in the early 1900s and introductions of both warm- and coolwater species, these lakes have supported important fisheries. Zinc, copper, cadmium, lead, mercury, and arsenic are of greatest concern especially in edible portions of fish flesh. To date, however, analysis of fish flesh has been completed with little replication (Rabe and Bauer 1977) or as a small component of a multifaceted project (Bennett and Bowles 1985).

Concentrations of heavy metals in fish flesh have not been adequately evaluated in the Coeur d' Alene system. Previous studies have examined fish composites where the entire fish was homogenized, thereby providing a mean whole-fish metal concentration not representative of the health risks from human consumption. Bennett and Bowles (1985) demonstrated that certain metals concentrate in specific organs. Therefore, only the fillets were tested in the Killarney Lake samples to determine risks from consumption. Also, mean values of a sample of fish flesh only show the "average" of the distribution and do not provide information on variation between individual fish. Sample sizes have been small and the representativeness of the sample has been questioned. Evaluation of the potential health hazards require analysis of the distribution and calculation of probabilities of encountering a fish with a concentration above an acceptable health level.

The purpose of this study was to design and execute pilot sampling of selected fish from Killarney Lake, Idaho, for heavy metal concentrations (zinc, copper, lead, cadmium, mercury, and arsenic). Analysis of results from this pilot study will enable us to assess the need for additional sampling. If more work is needed, then a statistically valid sampling effort for Killarney Lake could be designed. Specific objectives were:

OBJECTIVES

1. To conduct pilot sampling and analysis of fish flesh for selected heavy metals in Killarney Lake, Coeur d' Alene River system, Idaho;
2. To design a statistically valid sampling effort to describe the population parameters of heavy metal concentrations in fish flesh from Killarney Lake; and
3. To make recommendations for management and/or future research in the Coeur d' Alene River.

METHODS

Study Area

Fish samples were collected from Killarney Lake, Coeur d' Alene Lake system, Idaho (Figure 1). Killarney Lake, approximately 508 ha in surface area, is the second lake in a series of nine lakes immediately adjacent to the Coeur d' Alene River. The lakes flood annually during high spring runoff flows inundating vast expanses of emergent vegetation including *Scirpus* spp., *Equisetum* spp. and *Typha* spp. and other emergent wetland species. During low river flows, water flows out of Killarney Lake into the Coeur d' Alene River but river water generally flows into the lake at high river flows.

Collections

A total of ten each of largemouth bass (*Micropterus salmoides*), northern pike (*Esox lucius*), black crappie (*Pomoxis nigromaculatus*), brown bullhead (*Ictalurus nebulosus*), and yellow perch (*Perca flavescens*), were collected in Killarney Lake during fall 1989. These species represent the majority of fishes harvested by anglers in the lateral lakes. A majority of the fish were collected along the shoreline by nighttime electrofishing although a few were collected by gill netting. Total lengths and weights were recorded and scale samples were collected for aging each fish. A wide range of fish sizes were collected to be more representative of the total fish population and sizes of fish that could be creeled by anglers. Immediately following collection, fish were filleted and frozen at -5° F in locked freezers at the University of Idaho until thawing for analysis. Replicate flesh samples were cut from frozen fillets and processed identically to other samples. No fish organs and composite samples were

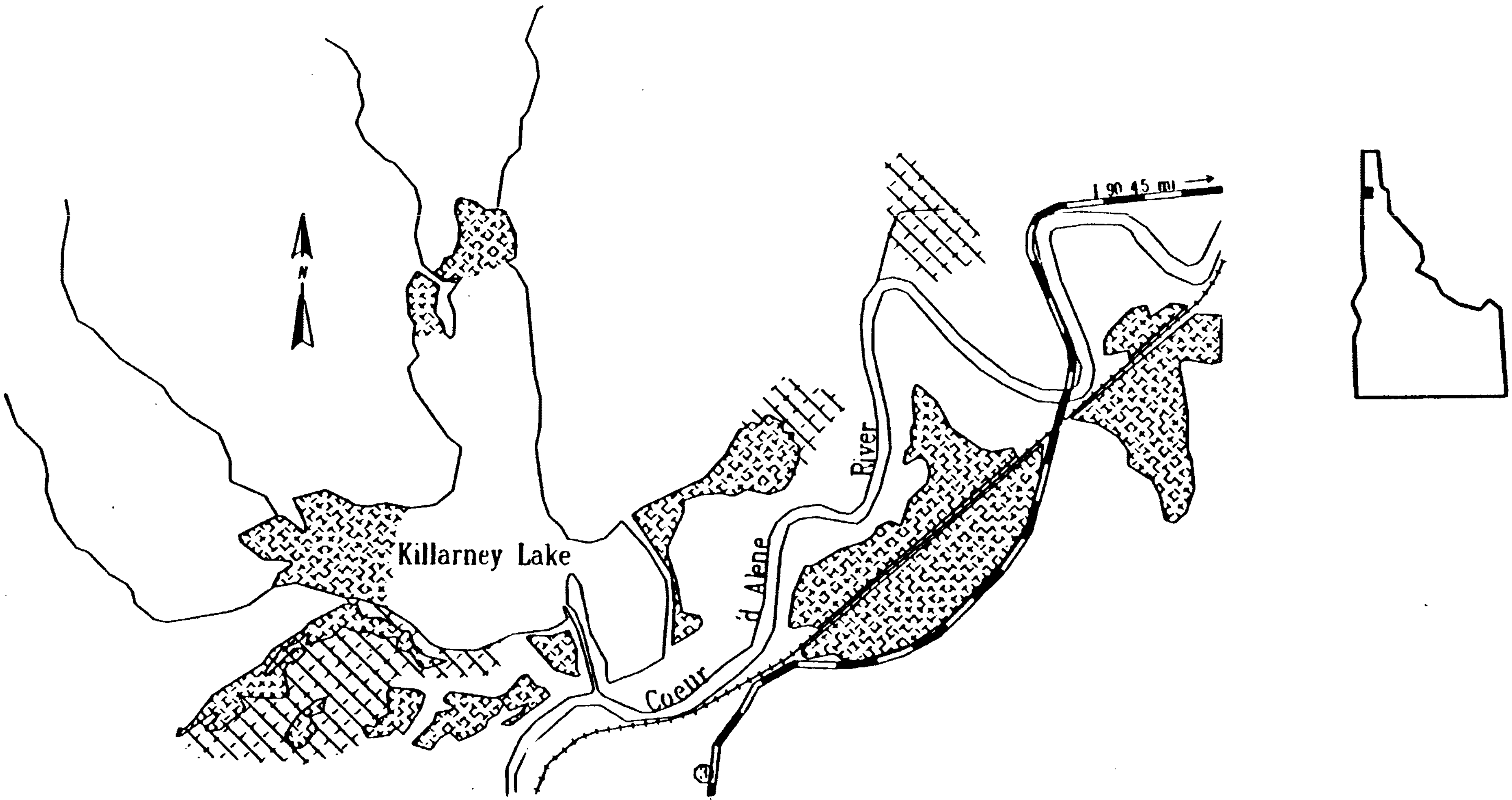

Figure 1. Map of Killarney Lake, one of the lateral lakes on the Coeur d' Alene River, near Harrison, Idaho.

analyzed in this study. Flesh samples representative of those possible for human consumption only were analyzed.

Digestion

Three grams of fish tissue were dissected from each fish fillet and digested with 6 ml of concentrated nitric acid. Samples were then placed in a microwave oven and baked 2 minutes plus an additional 30 seconds for each sample. Following baking, the digested samples are allowed to cool 20 minutes and then diluted to 13 ml with distilled water. Blanks (3 ml of distilled water and 6 ml of nitric acid) were also baked and diluted with the samples for use as controls during metal analysis.

Zinc-Copper Analyses

The digested samples were placed in the auto sampler tray of the Perkin Elmer Zeeman 5700 Atomic Absorption Spectrophotometer and measured by atomic absorption spectrophotometry. Standards and blanks were run initially and after every 10-15 samples for recalibration. Standards were mixed copper and zinc solutions of 2.0, 1.0, 0.5, and 0.1 mg/l concentrations.

Lead-Cadmium Analyses

Digestates were placed into the Graphite Furnace sampler tray of the Perkin Elmer Zeeman 5700 Atomic Absorption Spectrophotometer along with a blank and solutions of 0.10 mg/l cadmium or lead (standard), 0.2% nitric acid (HNO_3) (diluent), and 2 mg/ml ammonia hypophosphate ($NH_4H_2PO_4$) + 0.1 mg/ml magnesium nitrate ($Mg(NO_3)_2$) (matrix modifier). The single element program for lead or cadmium was selected and the appropriate Electrodeless Discharge lamp placed into operation. Lead analyses were done at 283.3 nm and the cadmium at 228.8 nm. Using a slit width of 0.07 nm, 0.010 ml of sample, 0.010 ml of diluent, and 0.010 ml of matrix modifier were simultaneously injected into the graphite furnace and the temperature program started. Both analyses were done versus standards of 0.05, 0.02, and 0.01 mg/l of the element of interest.

Mercury-Arsenic Analyses

Arsenic and mercury analyses were conducted using a mercury hydride system and a Perkin Elmer 360 Atomic Absorption Spectrometer. One half a milliliter of the digested sample was placed in a reaction vessel with 20 ml of 1.5% HCl. During operation, a solution of 3% $NaBH_4$ and 1% NaOH was injected into the reaction vessel generating arcsine or elemental mercury which was swept into the quartz tube. Standards of 0.50, 0.10, 0.05, and 0.01 mg/l arsenic and 0.005, 0.025, 0.010 mg/l mercury were run. The recorded deflections were measured and entered into a linear least squares regression program along with the sample ID, weight, volume, and any appropriate dilution factors to calculate the concentration in the original samples.

On January 9, 1990, 12 fish samples, including all the northern pike and samples 12 (largemouth bass) and 29 (black crappie), were tested at the Veterinary Sciences Laboratory at the University of Idaho. Three randomly selected tissue samples were taken from fish samples 1, 48, 12, and 29 and one sample each was taken from the remaining fish samples. To provide quality assurance and quality control on our analysis, 20 g each of fish samples 1, 12, 29, and 48, were sent to Laucks Laboratories, Seattle, Washington, to analyze for the same metals. Samples 1, 12, 29 and 48 were selected for further analysis for the following reasons: Sample 1, a northern pike, showed high concentrations of Cu (0.77 mg/l), sample 48, a northern pike, showed high concentrations of Zn (3.8 mg/l) and Cu at (0.41 mg/l), and sample 29, a black crappie, showed high concentrations of Zn (5.9 mg/l) and Hg (0.13 mg/l). The remaining northern pike samples were analyzed due to a broad difference in mercury and lead within the 10 samples.

We computed mean metal concentrations, variances, and 95% confidence intervals for each flesh sample. Variation in metals among the various species was compared by examining the coefficient of variation (standard deviation/mean) x 100. All metal concentrations are expressed in mg/kg wet weight. Tolerance limits for 99 and 95% confidence for 90 and 99% of the population were computed by (Lieberman 1958):

$$TL = M + (K)(SD)$$

where:

TL = Tolerance limits

M = Mean concentration (n = 10)

K = Tabular constant

SD = Standard deviation of metal concentration

Sample sizes required to describe the population mean of metal concentrations in fish flesh was assessed by (Zar 1984):

$$n = (s^2 (t^2_{a(2,n-1)} F_{B(1),(n-1,v)})/d^2$$

where:

n = Required sample size

s^2 = Sample variance

$t^2_{a(2,n-1)}$ = Square of tabular t - value

$F_{B(1),(n-1,v)}$ = One-tailed critical value from F distribution

d^2 = Square of acceptable difference in metals concentrations

Suggested maximum fish consumption quantities (kg/week) were computed by:

$$MAI = (MDI - MEDI)/ MMC$$

where:

MAI = Maximum allowable human consumption

MDI = Maximum dietary intake

MEDI = Mean dietary intake

MMC = Mean metal concentration

RESULTS

Zinc

Concentrations of zinc varied little among the five species of fishes sampled. Mean concentrations ranged from a high of 4.79 mg/kg (ppm) in yellow perch to a low of 2.95 mg/kg in brown bullheads (Figure 2). Similarly, variation among the individuals sampled was highest in yellow perch where confidence intervals were widest and coefficients of variation were about 32% (Table 1). Computed tolerance limits indicated that we were 99% confident that 99% of the fish populations sampled were below zinc concentrations that ranged from 15.02 mg/kg in yellow perch to 5.89 mg/kg in largemouth bass (Table 2). Replicated samples at Laucks Laboratory were similar but generally lower values than those determined at the UI Lab (Table 3).

Copper

Concentrations of copper were similar among species; northern pike, largemouth bass, brown bullheads, and yellow perch had nearly identical concentrations in flesh (Figure 3). Black crappies contained slightly lower concentrations. Mean copper concentrations ranged from 0.302 mg/kg in brown bullheads to 0.265 mg/kg in black crappie. Variation in concentration was highest in northern pike which accounted for the wide confidence intervals and a coefficient of variation of 59% (Table 1). Tolerance limits indicated that we were 99% confident that 99% of the fish populations sampled were below 1.51 mg/kg copper (Table 2). Replicated analyses were variable and not similar between labs (Table 3). For example, black crappie No. 29 had flesh levels that varied from 0.22 to 3.2 mg/kg. Multiple analyses from other fish were less variable.

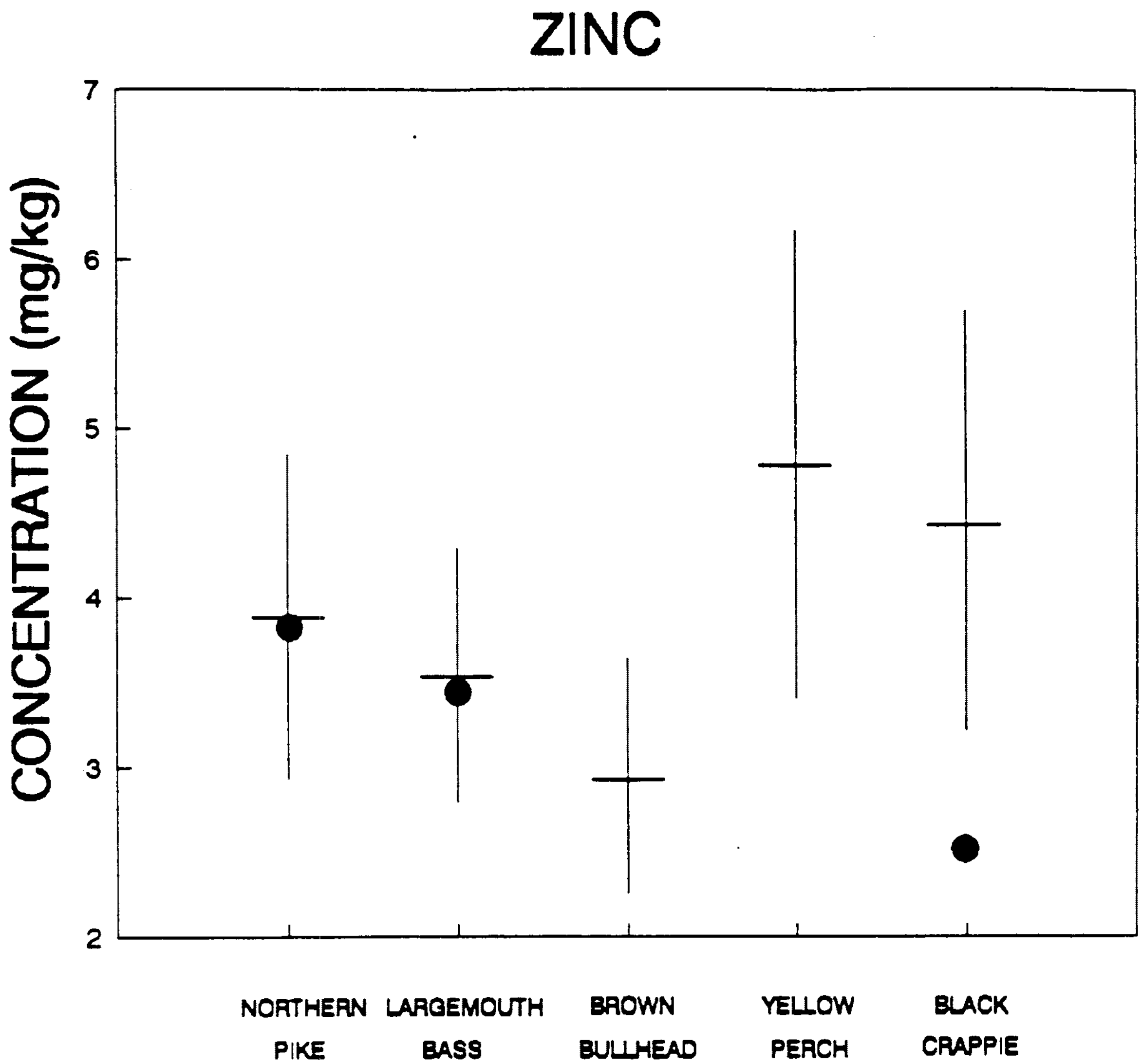

Figure 2. Mean (horizontal line) and 95% confidence intervals for concentrations of zinc in five species of fish (n=10) from Killarney Lake, Idaho. Black circles represent estimated zinc concentrations for a single flesh sample (n=1) conducted by a second laboratory for quality assurance and quality control.

TABLE 1. CONCENTRATIONS OF HEAVY METAL (mg/kg wet weight) IN FISHES FROM KILLARNEY LAKE ANALYZED AT THE VET SCIENCE LABORATORY, UNIVERSITY OF IDAHO.

FISH NO	SPECIES	LENGTH	WEIGHT	AGE	Zn	Cu	Pb	Cd	Hg	As
1	Northern Pike	464	595	2	3.20	0.77	0.03	0.02	<.04	<.04
2	Northern Pike	457	505	2	3.10	0.44	0.03	0.03	<.04	<.04
3	Northern Pike	437	519	2	3.00	0.32	0.07	0.02	<.04	<.04
4	Northern Pike	395	347	2	3.70	0.23	0.03	0.03	<.04	<.04
5	Northern Pike	438	450	2	4.00	0.22	0.03	0.02	<.04	<.04
46	Northern Pike	480	650	2	3.90	0.27	0.21	0.01	0.10	<.02
47	Northern Pike	498	725	2	4.00	0.13	0.05	0.01	0.06	<.02
48	Northern Pike	529	875	2	5.69	0.19	0.04	0.01	0.05	<.02
49	Northern Pike	522	925	2	4.91	0.26	0.03	0.01	0.10	<.02
50	Northern Pike	598	1350	2	3.36	0.25	0.08	0.01	0.12	<.02
MEAN		482	694		3.89	0.31	0.06	0.04	0.05	
COEFFICIENT OF VARIATION					21.89	59.04	93.61	138.26	75.50	
6	Largemouth Bass	498	2638	8	3.30	0.12	0.02	0.02	0.05	<.04
7	Largemouth Bass	358	750	5	3.60	0.30	0.02	0.03	<.04	<.04
8	Largemouth Bass	404	1189	6	3.80	0.31	0.01	0.01	0.05	<.04
9	Largemouth Bass	395	1093	6	3.50	0.41	0.02	0.02	<.04	<.04
10	Largemouth Bass	322	552	5	3.20	0.30	0.01	0.01	<.04	<.04
11	Largemouth Bass	324	595	5	3.60	0.28	0.02	0.01	0.05	<.04
12	Largemouth Bass	319	526	5	3.80	0.41	0.01	0.02	0.20	<.04
13	Largemouth Bass	431	1382	7	2.80	0.26	0.01	0.02	0.08	<.04
14	Largemouth Bass	418	1326	7	3.80	0.37	0.01	0.02	<.04	<.04
15	Largemouth Bass	352	717	5	4.00	0.31	0.01	0.02	<.04	<.04
MEAN		382	1077		3.54	0.31	0.01	0.02	0.05	
COEFFICIENT OF VARIATION					10.07	27.30	36.89	35.14	104.88	
16	Brown Bullhead	276	311		3.80	0.56	0.06	0.03	<.04	<.04
17	Brown Bullhead	255	235		2.40	0.19	0.03	0.03	0.09	<.04
18	Brown Bullhead	227	166		3.10	0.50	0.09	0.05	<.04	0.06
19	Brown Bullhead	248	206		2.50	0.16	0.09	0.03	<.04	<.04
20	Brown Bullhead	210	127		2.60	0.17	0.03	0.02	<.04	<.04
21	Brown Bullhead	193	90		2.80	0.28	0.08	0.03	0.05	<.02
22	Brown Bullhead	181	78		3.70	0.35	0.18	0.05	<.04	<.02
23	Brown Bullhead	203	106		2.70	0.18	0.24	0.05	0.06	<.02
24	Brown Bullhead	253	204		2.30	0.38	0.03	0.04	<.04	<.02
25	Brown Bullhead	221	157		3.60	0.25	1.00	0.03	<.04	<.02
MEAN		227	168		2.95	0.30	0.18	0.04	0.03	
COEFFICIENT OF VARIATION					19.13	47.13	161.28	29.86	72.31	

TABLE 1. Cont.

FISH NO	SPECIES	LENGTH	WEIGHT	AGE	Zn	Cu	Pb	Cd	Hg	As
26	Black Crappie	297	449	8	2.10	0.25	0.04	0.04	0.06	<.02
27	Black Crappie	267	347	6	3.80	0.38	0.02	0.03	0.16	<.02
28	Black Crappie	208	146	5	3.20	0.29	0.03	0.03	0.07	<.02
29	Black Crappie	236	218	5	5.90	0.26	0.06	0.03	0.13	<.02
7	Black Crappie	211	155	3	5.60	0.23	0.05	0.02	<.04	<.02
31	Black Crappie	211	152	4	4.20	0.20	0.04	0.03	<.04	<.02
32	Black Crappie	221	165	5	4.30	0.29	0.01	0.04	0.05	<.02
33	Black Crappie	197	117	4	4.60	0.27	0.02	0.03	<.04	<.02
34	Black Crappie	212	146	5	4.20	0.24	0.01	0.02	0.05	<.02
35	Black Crappie	198	116	5	6.70	0.24	0.02	0.03	0.07	<.02
MEAN		226	201		4.46	0.27	0.03	0.03	0.07	
COEFFICIENT OF VARIATION					30.05	18.42	56.66	22.22	72.25	
36	Yellow Perch	171	59	5	4.00	0.23	0.10	0.01	0.08	<.02
37	Yellow Perch	202	95	5	4.40	0.33	0.04	0.03	0.39	<.02
38	Yellow Perch	165	52	4	5.70	0.21	0.16	0.04	0.05	<.02
39	Yellow Perch	161	53	3	3.40	0.24	0.01	0.02	<.04	<.02
40	Yellow Perch	164	49	4	3.40	0.20	0.02	0.02	0.07	<.02
41	Yellow Perch	163	48	4	3.60	0.33	0.02	0.04	0.07	<.02
42	Yellow Perch	155	43	4	4.90	0.24	0.11	0.04	0.05	<.02
43	Yellow Perch	143	31	4	7.90	0.25	0.71	0.04	0.10	<.02
44	Yellow Perch	146	36	4	6.80	0.71	0.10	0.05	0.08	<.02
45	Yellow Perch	153	40	4	3.80	0.32	0.07	0.87 /3	<.04	<.02
MEAN		162	51		4.79	0.31	0.13	0.12	0.09	
COEFFICIENT OF VARIATION					32.33	49.04	155.28	228.63	115.59	

/1 Metal analysis was ran twice on fish numbers 1 and 2 due to abnormally high readings for Cd. The readings from the second analysis are used in this table and the statistics.
/2 Metal analysis was ran twice on fish numbers 46-50 due to abnormally high readings for Hg and Pb. The readings from the second analysis are used in this table and the statistics.
/3 Considered an outlier, not used in statistical calculations.

Table 2. Comparison of action levels, means, variances, standard deviations and tolerance limits (95% and 99% confident that 90% and 99% of the population is below a concentration) for fishes from Killarney Lake, Idaho.

Species	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	Comments
Northern Pike	Zn	Cu	Pb	Cd	Hg	
action	45.00	20.00	3.50	0.07	1.00	
mean	3.89	0.31	0.06	0.02	0.05	
variance	0.72	0.03	0.00	0.00	0.00	
std dev	0.85	0.18	0.06	0.01	0.04	
tolerance limits	5.89	0.74	0.19	0.03	0.15	95% confident that 90% of the population is below this level.
	6.48	0.86	0.23	0.04	0.17	99% confident that 90% of the population is below this level.
	9.50	1.51	0.43	0.07	0.32	99% confident that 99% of the population is below this level.
Largemouth Bass	Zn	Cu	Pb	Cd	Hg	
action	45.00	20.00	3.50	0.07	1.00	
mean	3.54	0.31	0.01	0.02	0.05	
variance	0.13	0.01	0.00	0.00	0.00	
std dev	0.36	0.08	0.01	0.01	0.06	
tolerance limits	4.38	0.50	0.03	0.03	0.18	95% confident that 90% of the population is below this level.
	4.63	0.56	0.03	0.04	0.22	99% confident that 90% of the population is below this level.
	5.89	0.86	0.05	0.06	0.42	99% confident that 99% of the population is below this level.
Brown Bullhead	Zn	Cu	Pb	Cd	Hg	
action	45.00	20.00	3.50	0.07	1.00	
mean	2.95	0.30	0.18	0.04	0.03	
variance	0.32	0.02	0.09	0.00	0.00	
std dev	0.56	0.14	0.30	0.01	0.02	
tolerance limits	4.28	0.64	0.88	0.06	0.09	95% confident that 90% of the population is below this level.
	4.67	0.74	1.08	0.07	0.11	99% confident that 90% of the population is below this level.
	6.68	1.24	2.13	0.11	0.20	99% confident that 99% of the population is below this level.

Table 2. Cont.

	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	
Black Crappie	Zn	Cu	Pb	Cd	Hg	
action	45.00	20.00	3.50	0.07	1.00	
mean	4.46	0.27	0.03	0.03	0.07	
variance	1.80	0.00	0.00	0.00	0.00	
std dev	1.34	0.05	0.02	0.01	0.05	
tolerance limits	7.62	0.38	0.07	0.05	0.18	95% confident that 90% of the population is below this level.
	8.54	0.41	0.08	0.05	0.21	99% confident that 90% of the population is below this level.
	13.31	0.59	0.14	0.07	0.38	99% confident that 99% of the population is below this level.
Yellow Perch	Zn	Cu	Pb	Cd	Hg	
action	45.00	20.00	3.50	0.07	1.00	
mean	4.79	0.31	0.13	0.03	0.09	
variance	2.40	0.02	0.04	0.00	0.01	
std dev	1.55	0.15	0.21	0.01	0.11	
tolerance limits	8.44	0.66	0.62	0.06	0.35	95% confident that 90% of the population is below this level.
	9.51	0.76	0.77	0.07	0.42	99% confident that 90% of the population is below this level.
	15.02	1.30	1.51	0.12	0.80	99% confident that 99% of the population is below this level.

TABLE 3. COMPARISONS OF REPLICATE TISSUE SAMPLE ANALYSIS FOR HEAVY METAL CONCENTRATIONS BETWEEN LAUCKS LABORATORIES (L.L.), SEATTLE, WASHINGTON AND THE VETERINARY SCIENCES LABORATORY, UNIVERSITY OF IDAHO.

FISH NO.	SPECIES		Zn	Cu	Pb	Cd	Hg	As
1	Original reading		3.20	0.77	0.03	0.15	<0.04	<0.04
		1	4.90	0.15	0.05	0.02	0.07	<0.04
	Northern Pike	2	3.50	0.22	0.02	0.01	<0.04	0.10
		3	4.60	0.63	0.04	0.02	<0.04	0.18
	MEAN	/1	4.05	0.44	0.04	0.05	0.07	0.14
	L.L.		3.8	<1	<.5	<.5	0.2	<.5
48	Original reading		5.69	0.19	0.40	0.01	0.05	<0.02
		1	4.00	0.28	0.03	0.01	0.06	<0.04
	Northern Pike	2	4.80	0.15	0.04	0.02	<0.04	0.18
		3	4.20	0.15	0.05	0.01	0.06	0.10
	MEAN		4.67	0.19	0.13	0.01	0.06	0.14
	L.L.		3.8	<1	<.5	<.5	0.1	<.5
12	Original reading		3.80	0.41	0.01	0.02	0.20	<0.04
		1	3.70	0.31	0.01	0.02	0.11	<0.04
	Largemouth Bass	2	3.60	0.67	0.04	0.01	0.12	0.09
		3	3.20	0.16	0.02	0.06	0.14	0.09
	MEAN		3.58	0.39	0.02	0.03	0.12	0.09
	L.L.		3.4	2	<.5	<.5	0.1	<.5
29	Original reading		5.90	0.26	0.06	0.03	0.13	<0.02
		1	5.40	0.22	0.03	0.02	<0.04	0.06
	Black Crappie	2	5.40	3.20	0.06	0.02	0.04	0.09
		3	4.80	1.59	0.06	0.03	<.04	0.19
	MEAN		5.38	1.32	0.05	0.03	0.04	0.11
	L.L.		2.5	<1	<.5	<.5	0.1	<.5

/1 Means calculated without levels below the detection limits.

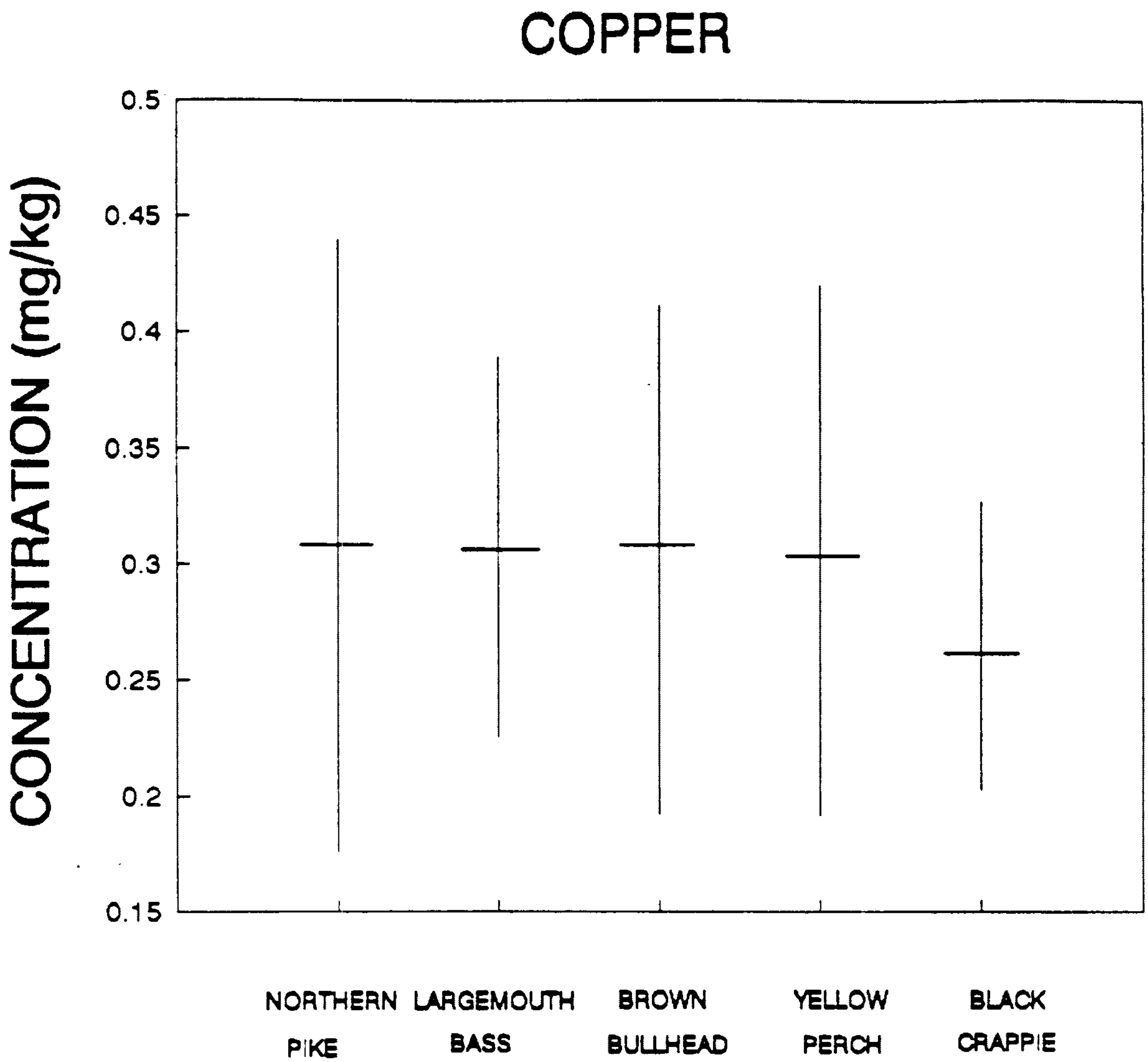

Figure 3. Mean (horizontal line) and 95% confidence intervals for concentrations of copper in five species of fish (n=10) from Killarney Lake, Idaho. No black circles are shown because values from a second laboratory were on a single flesh sample (n=1) were below detection limits.

Lead

Mean concentrations of lead were highest in brown bullheads (0.183 mg/kg) followed closely by yellow perch (0.13), while lowest concentrations were found in largemouth bass (0.014 mg/kg) (Figure 4). Lead variability was also highest in brown bullheads and lowest in largemouth bass (Table 2). Coefficients of variation in brown bullheads and yellow perch exceeded 155% (Table 1). Confidence intervals (95%) of yellow perch and bullheads overlapped those of other species. Calculated tolerance limits suggested that we were 99% confident that 99% of the fish populations sampled were below 2.13 mg/kg of lead (Table 2). Replicated samples from the UI lab indicated close agreement among readings (Table 3). Results from Laucks Lab had much higher detection limits and therefore, were not really comparable.

Cadmium

Mean cadmium concentrations ranged from 0.02 to 0.04 mg/kg. Mean concentrations were highest in brown bullheads and generally similar among black crappies, yellow perch, largemouth bass, and northern pike (Figure 5). Variability was highest in northern pike as evidenced by the 47.1% coefficient of variation (Table 1). Tolerance limits indicated that we were 99% confident that 99% of the fish populations sampled were below 0.12 mg/kg cadmium (Table 2). Cadmium determinations were generally similar among readings on the same flesh sample (Table 3). One exception was the determination on northern pike No. 1 which initially indicated a flesh concentration of 0.15 mg/kg, whereas three additional readings on the same fillet were consistently lower (0.02-0.01 mg/kg). Detections limits from Laucks Lab for cadmium of 0.5 mg/kg were high relative to those from the UI.

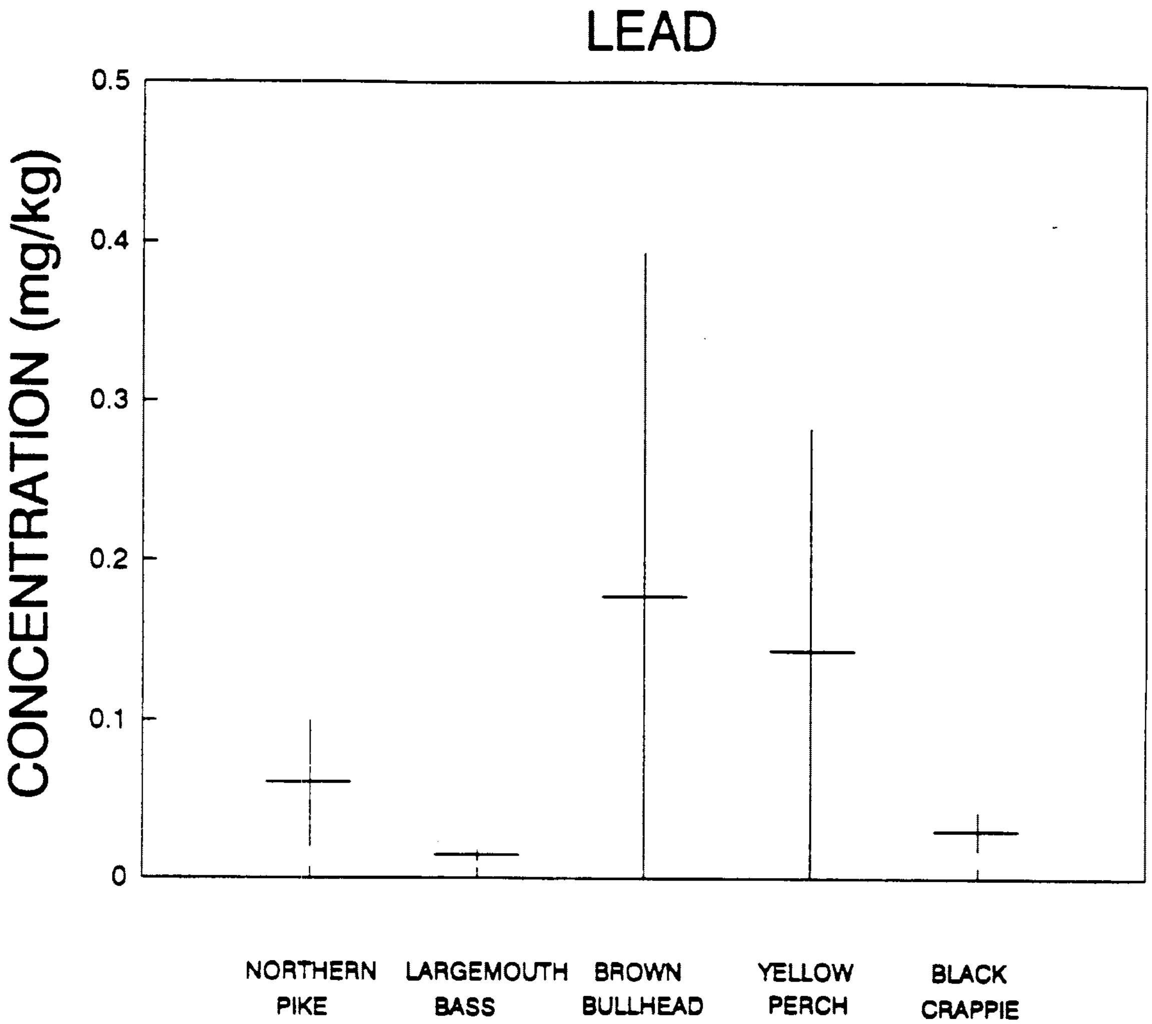

Figure 4. Mean (horizontal line) and 95% confidence intervals for concentrations of lead in five species of fish (n=10) from Killarney Lake, Idaho. No black circles are shown because values from a second laboratory were on a single flesh sample (n=1) were below detection limits.

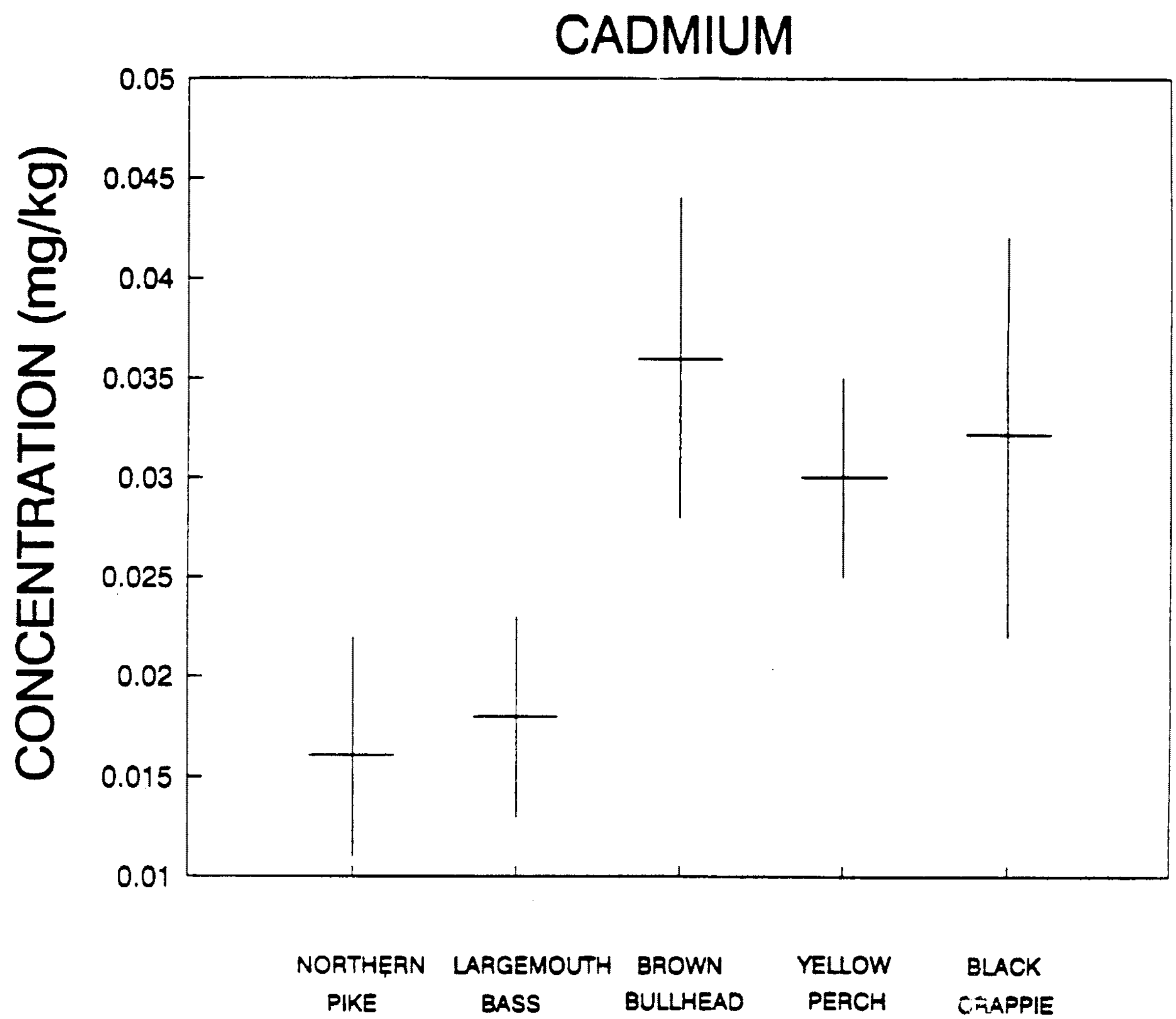

Figure 5. Mean (horizontal line) and 95% confidence intervals for concentrations of cadmium in five species of fish (n=10) from Killarney Lake, Idaho. No black circles are shown because values from a second laboratory were on a single flesh sample (n=1) were below detection limits.

Mercury

Mean concentrations ranged from 0.034 to 0.093 mg/kg; concentrations were highest in yellow perch and lowest in brown bullheads but not statistically different as 95% confidence intervals overlapped (Figure 6). Mean concentrations of mercury were intermediate and identical for largemouth bass and northern pike. Variation in mercury concentrations were highest in yellow perch (CV = 116%) followed by largemouth bass (CV = 104%; Table 1). Tolerance limits for mercury for 99% confidence for 99% of the fish populations were highest in yellow perch (TL = 0.80) and lowest in brown bullheads (Table 2). In general, mercury concentrations determined by Laucks Lab were higher than those from the UI Lab.

Arsenic

From the first series of analysis, concentrations of arsenic were below detection limits (<0.04 mg/kg) for all species sampled except for one brown bullhead sample (0.06 mg/kg; Table 1). Because of this, statistical comparisons on these samples could not be made. Random tissue samples from four of the same fish (1, 12, 29, 48), also analyzed at the University of Idaho, yielded values that ranged as high as 0.19 mg/kg (Table 3). Of all the metals analyzed, results from the arsenic analyses were most variable among the replicate fillets examined. Detection limits from Laucks Lab (<0.5 mg/kg) were considerably higher than those from the UI (<0.04 mg/kg).

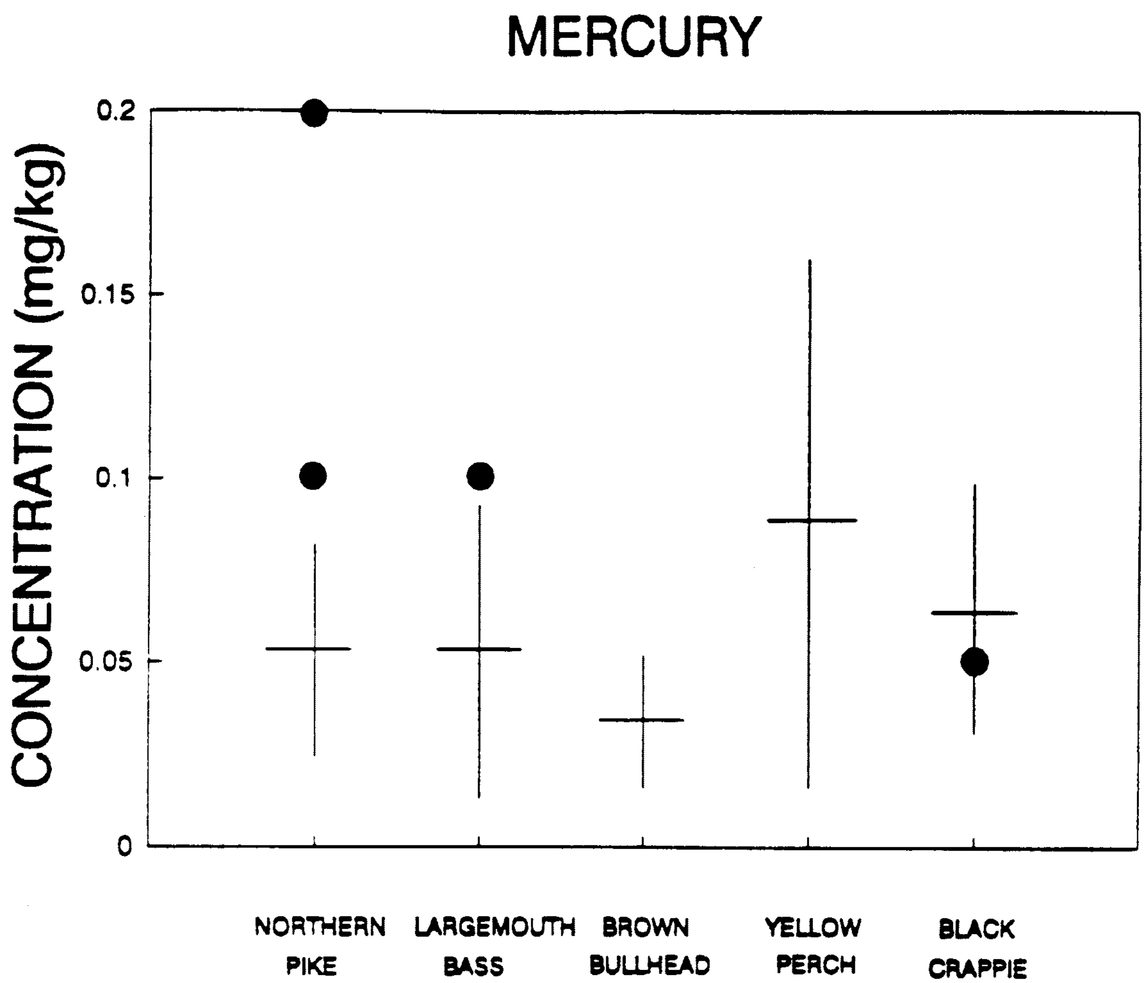

Figure 6. Mean (horizontal line) and 95% confidence intervals for concentrations of mercury in five species of fish (n=10) from Killarney Lake, Idaho. Black circles represent estimated mercury concentrations for a single flesh sample (n=1) conducted by a second laboratory for quality assurance and quality control.

Body Sizes

Concentrations of metals tested varied by size of fish in three species. Comparisons were made for each species collected for zinc, copper, cadmium, lead, and mercury. Three significant ($p<0.05$) correlations were found. Copper decreased significantly ($p=0.02$) in largemouth bass with increased weight and length (Figure 7). In contrast, concentrations of mercury increased significantly ($p=0.001$) with increases in both length and weight of yellow perch (Figure 8). Concentrations of zinc decreased significantly ($p=0.05$) with increased body length and weight of black crappie (Figure 9).

Quality Assurance/Quality Control

Concentrations of metals in fish flesh obtained from the UI VET Science Laboratory were closely comparable to those results obtained by Laucks Laboratories (Figures 2-7; Table 3). Detection limits were different between laboratories. In general, concentrations measured by Laucks Laboratories generally fell within the 95% confidence intervals of the samples analyzed at the U of I but were consistently lower than those measured for the same species except for mercury in largemouth bass which was higher. We tested multiple samples from the same fish fillet to examine how representative our samples were to the total fillet (Table 3). Concentrations of metals varied slightly depending on location in the fillet but generally were within 95% confidence intervals (Figures 10-14). The outlier of cadmium determination on northern pike No. 1 is clearly evident (Figure 13). No consistent trends

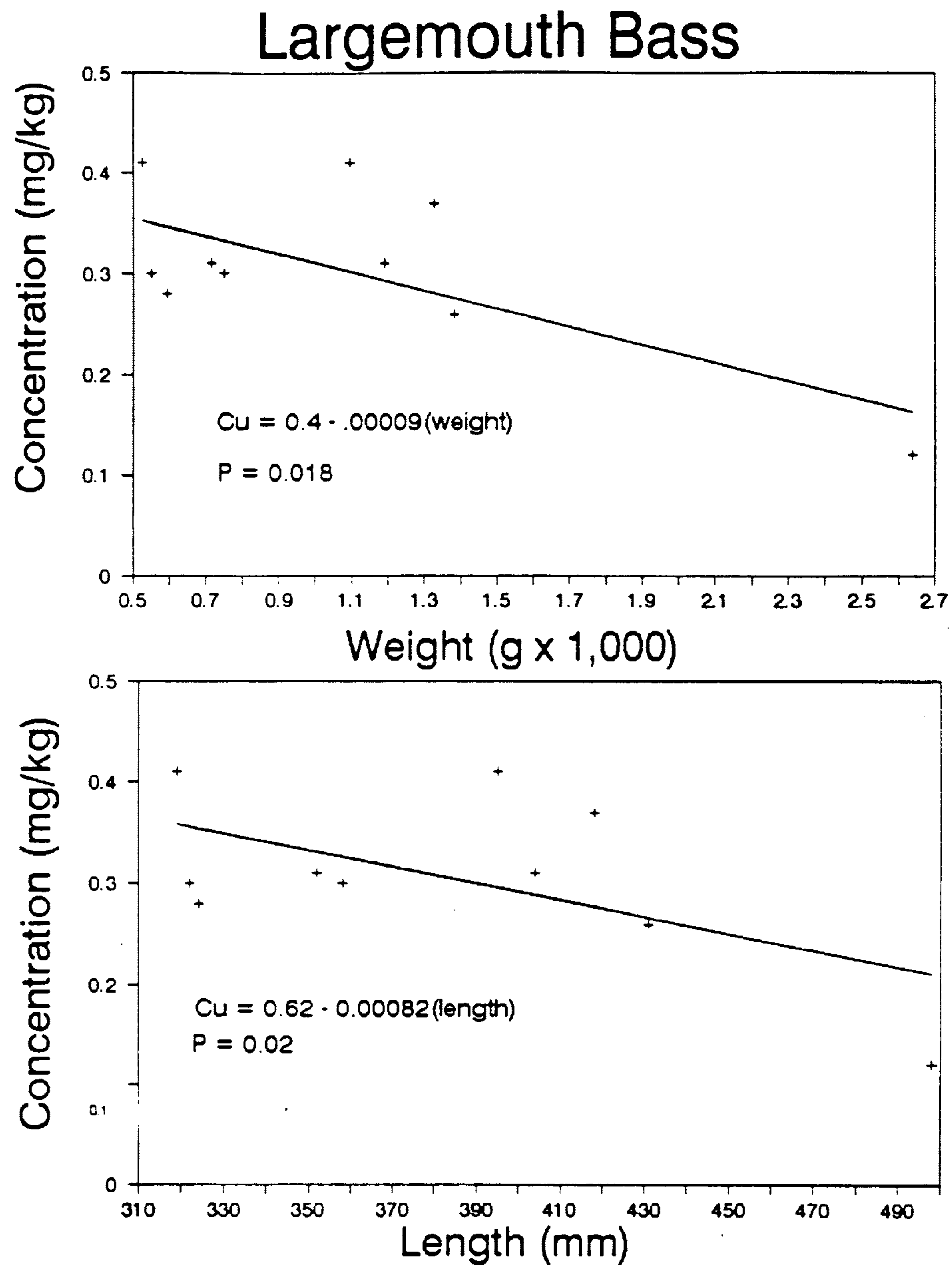

Figure 7. Linear relationships between concentration of copper in largemouth bass flesh as a function of body size.

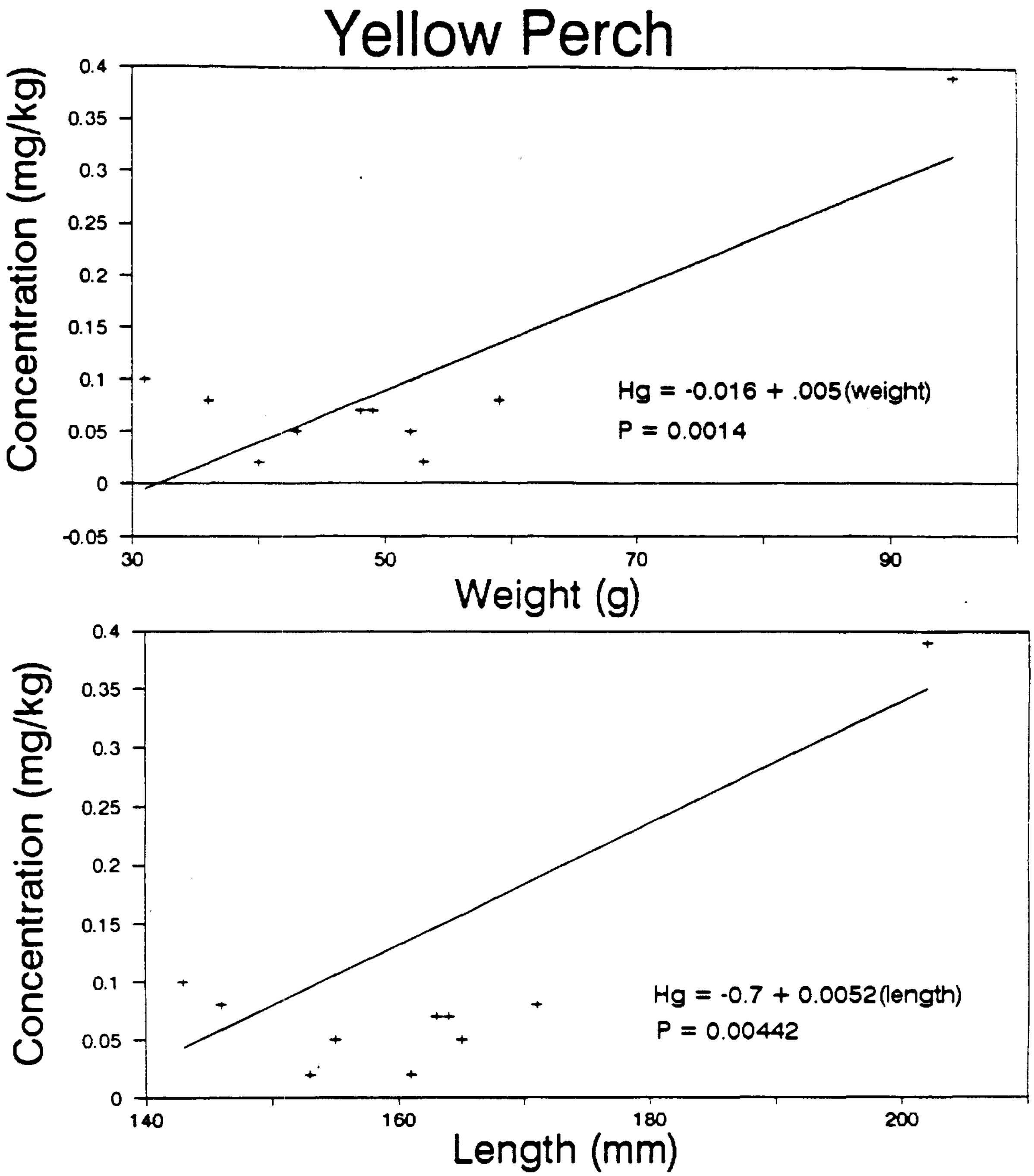

Figure 8. Linear relationships between concentration of mercury in yellow perch flesh as a function of body size.

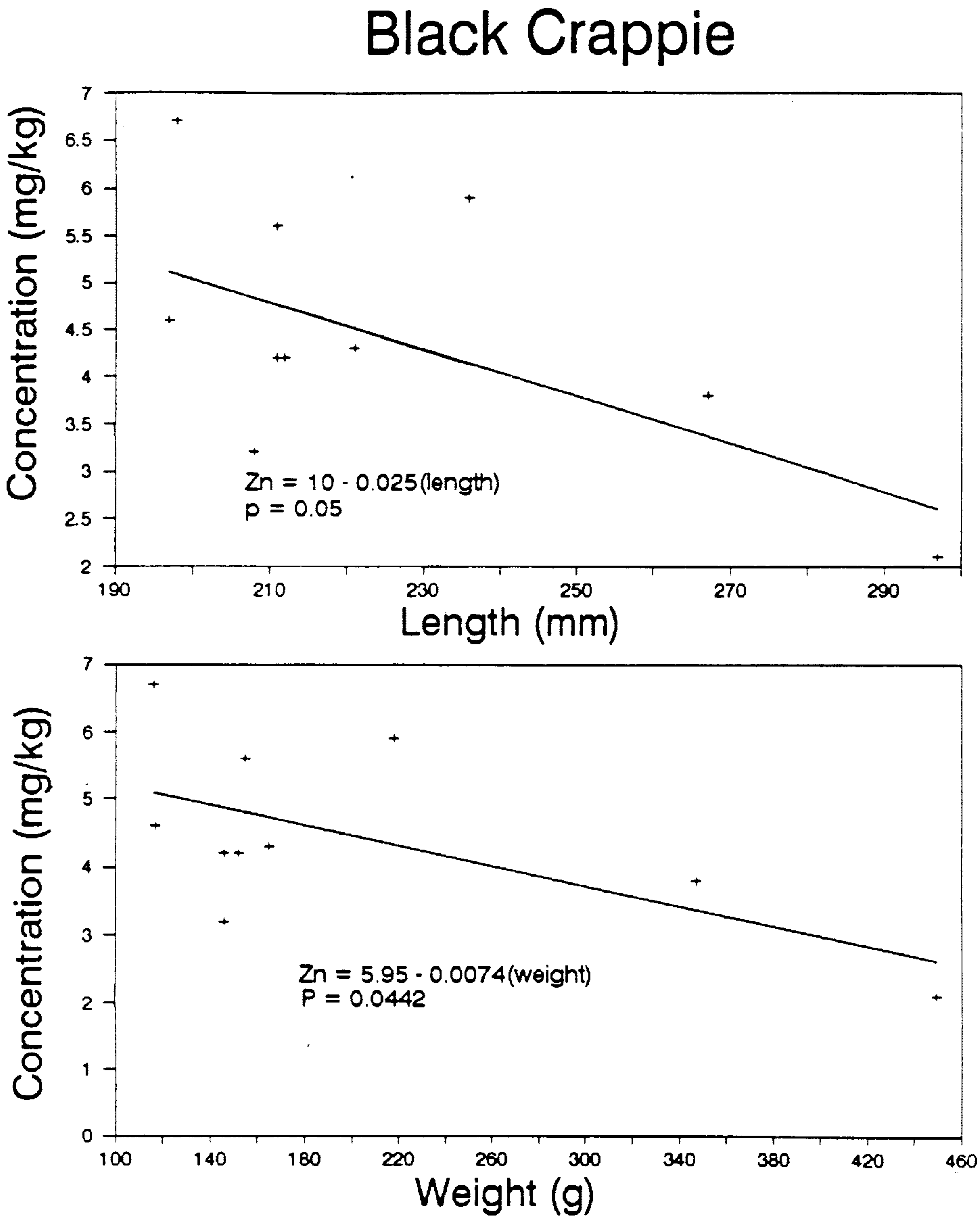

Figure 9. Linear relationships between concentration of zinc in yellow perch flesh as a function of body size.

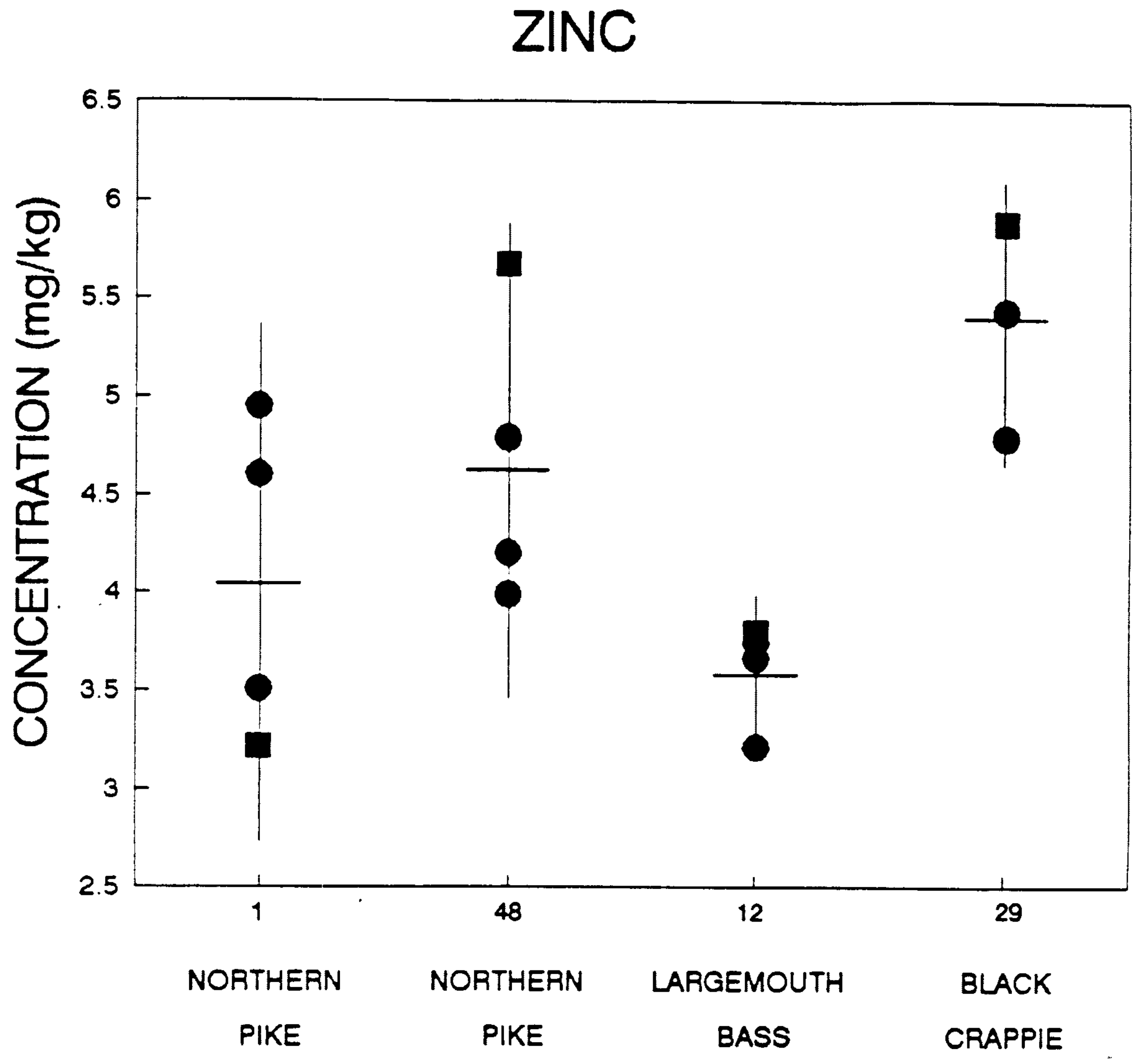

Figure 10. Comparison of replicate analyses from the same fish fillet. Squares represent the value of the initial determination while the circles represent zinc from randomly selected tissue samples. Mean concentration (horizontal bar) and 95% confidence intervals are shown for the four samples.

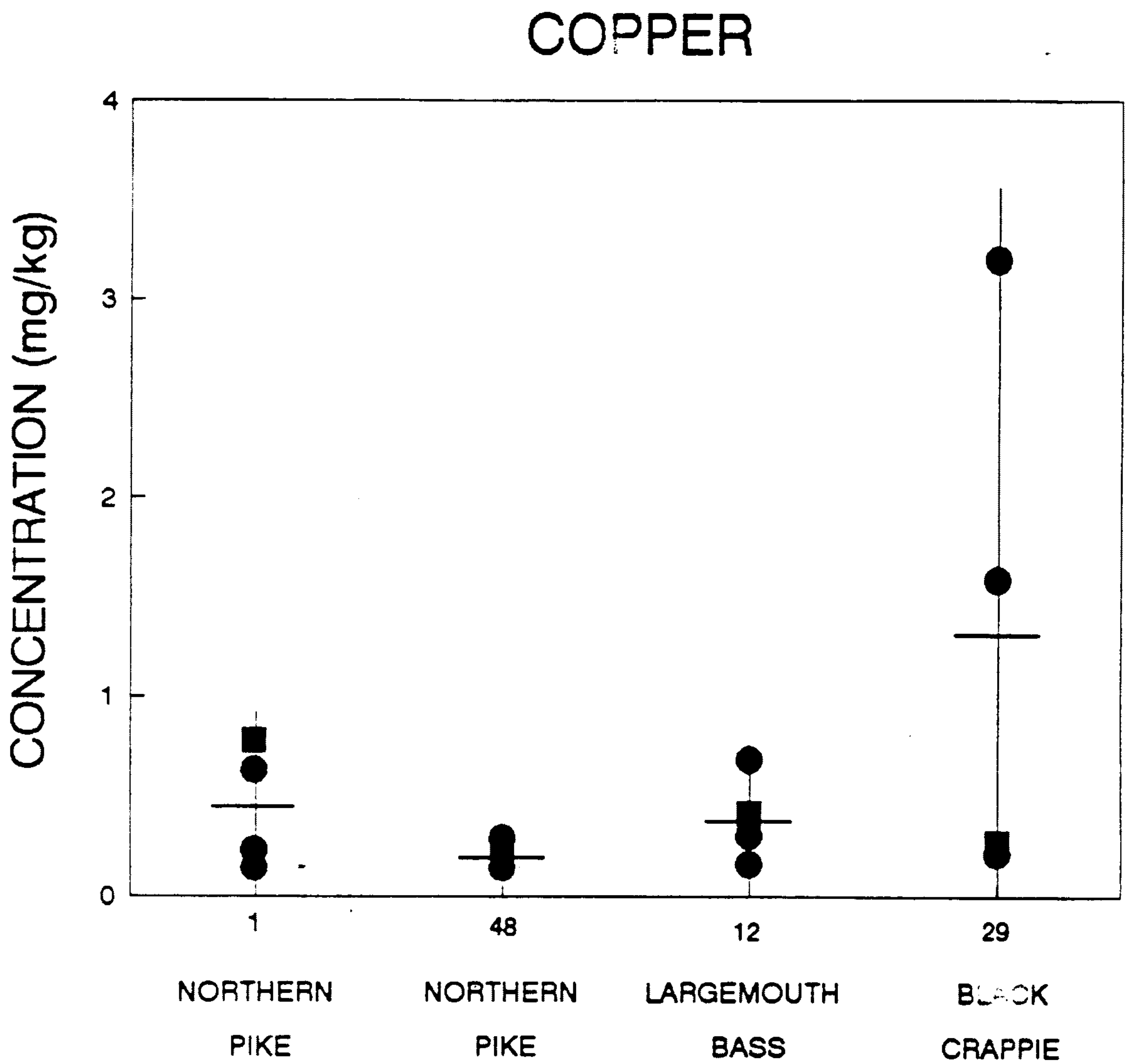

Figure 11. Comparison of replicate analyses from the same fish fillet. Squares represent the value of the initial determination while the circles represent copper from randomly selected tissue samples. Mean concentration (horizontal bar) and 95% confidence intervals are shown for the four samples.

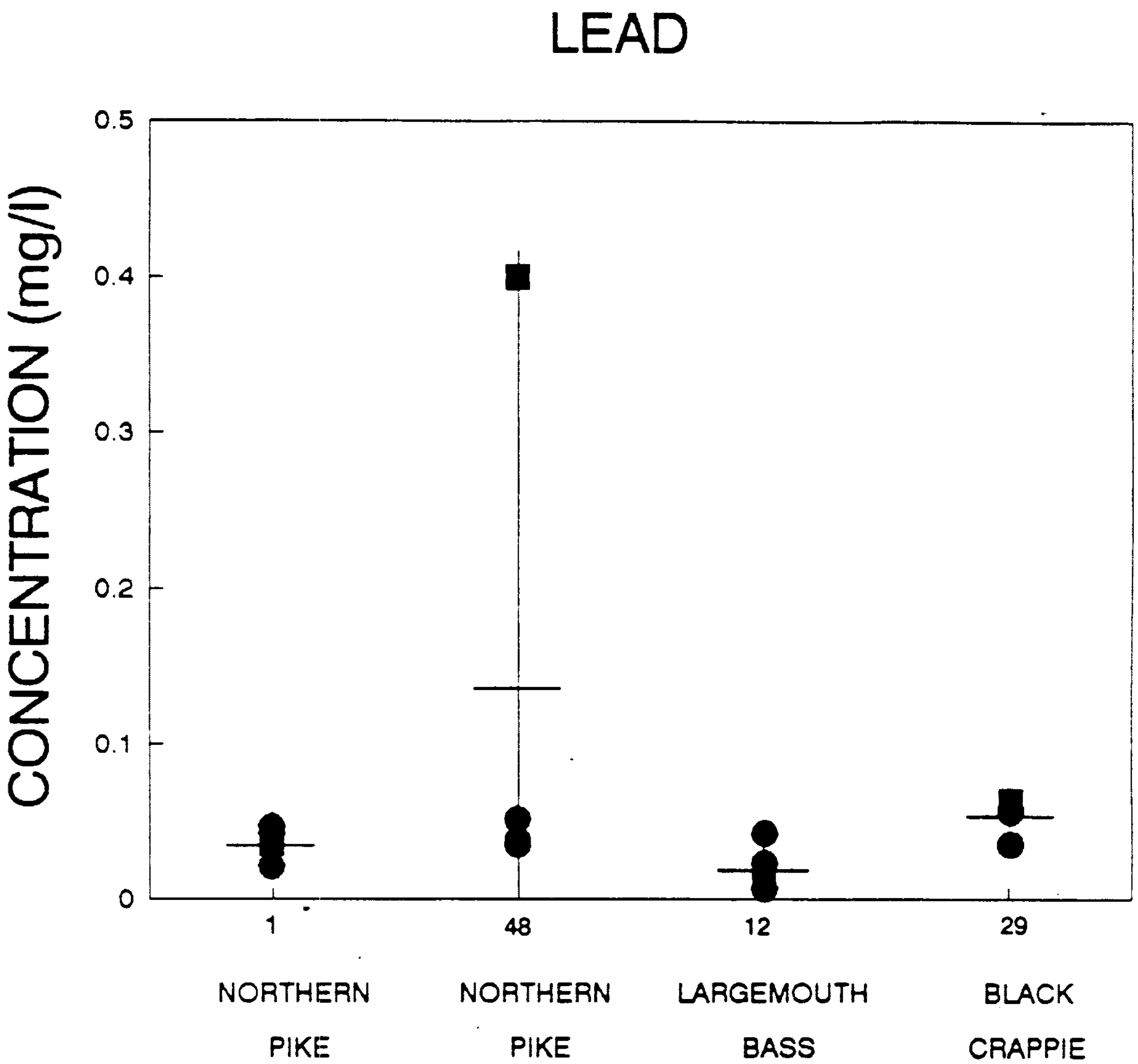

Figure 12. Comparison of replicate analyses from the same fish fillet. Squares represent the value of the initial determination while the circles represent lead from randomly selected tissue samples. Mean concentration (horizontal bar) and 95% confidence intervals are shown for the four samples.

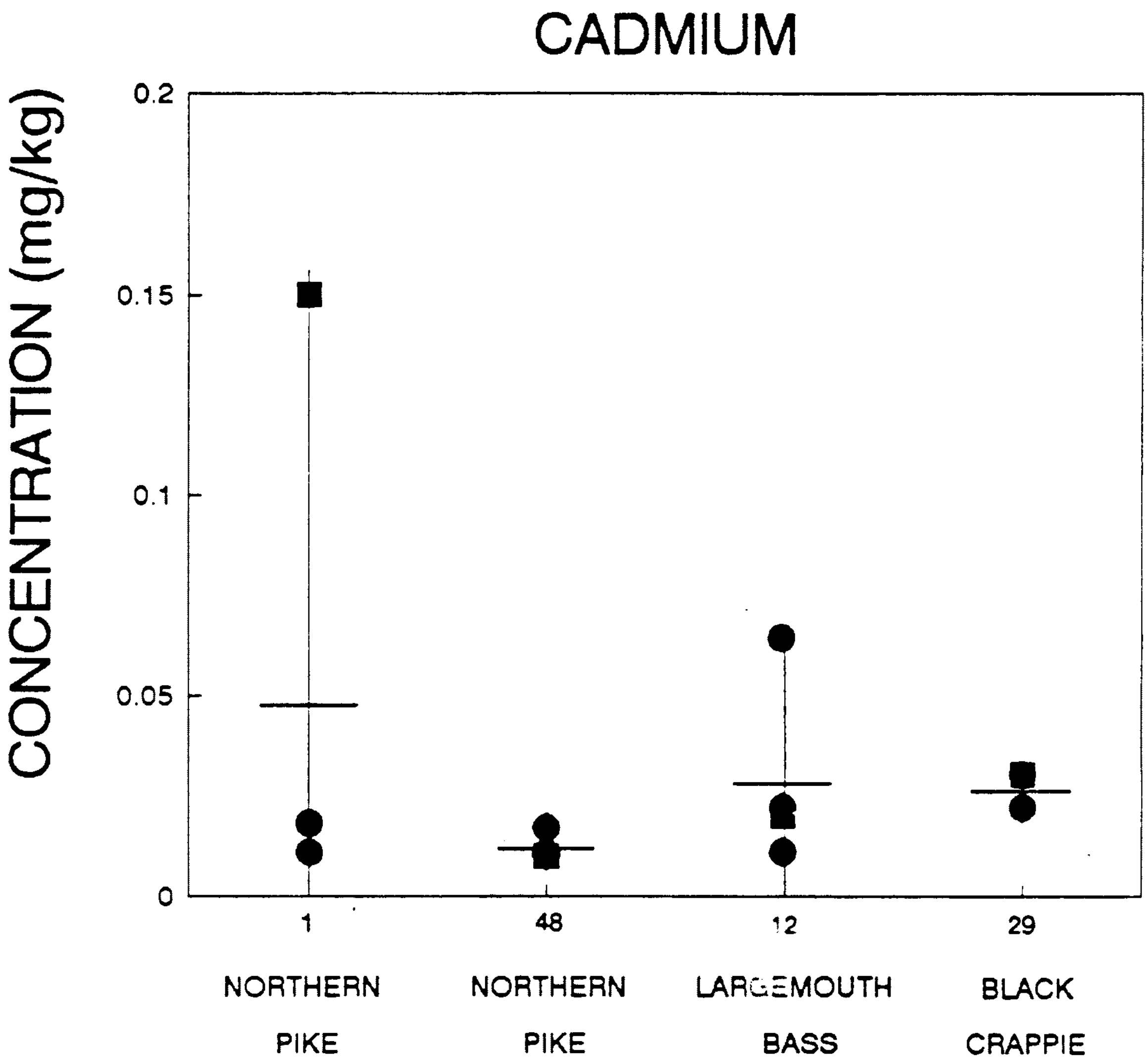

Figure 13. Comparison of replicate analyses from the same fish fillet. Squares represent the value of the initial determination while the circles represent cadmium from randomly selected tissue samples. Mean concentration (horizontal bar) and 95% confidence intervals are shown for the four samples.

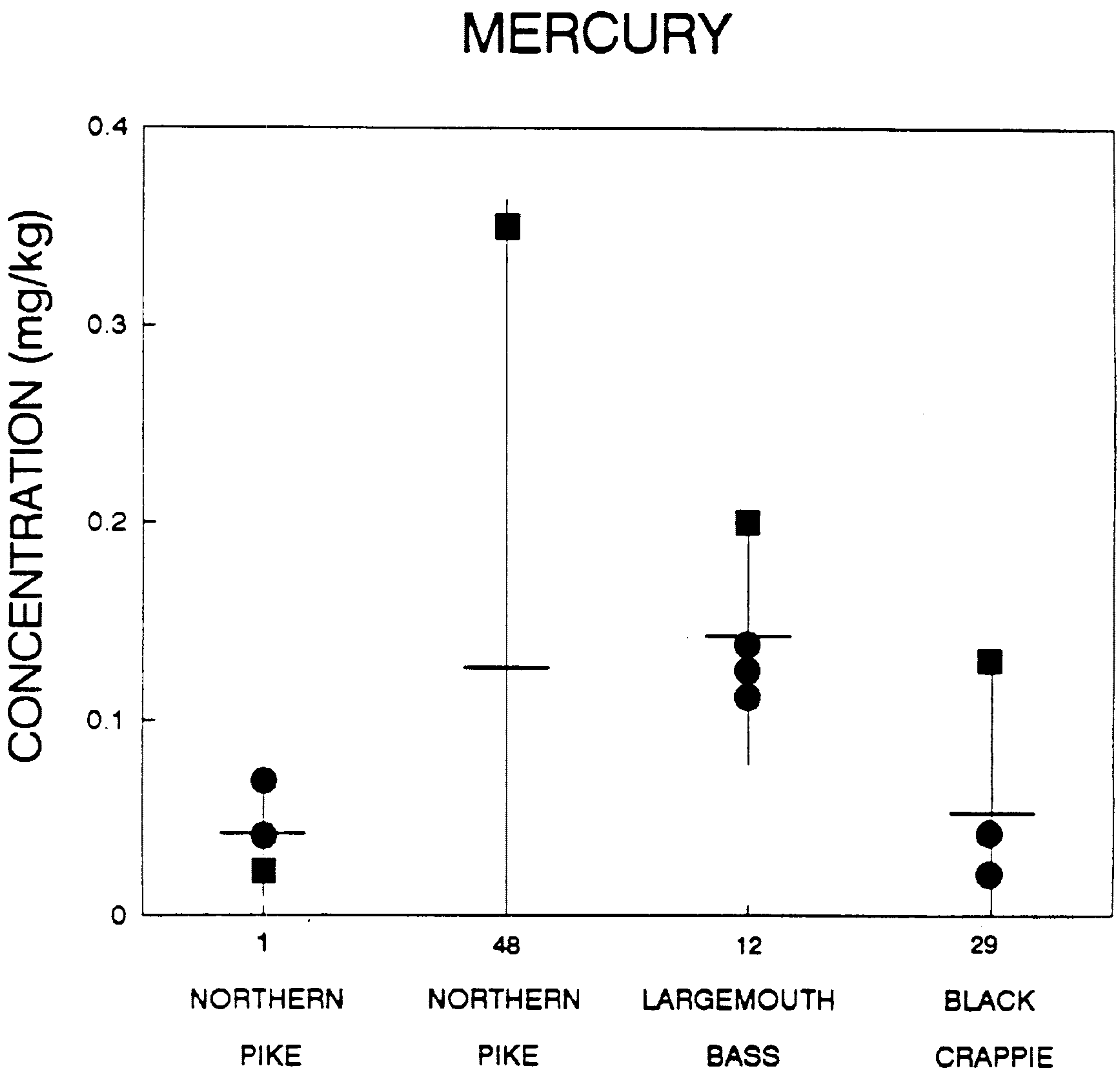

Figure 14. Comparison of replicate analyses from the same fish fillet. Squares represent the value of the initial determination while the circles represent mercury from randomly selected tissue samples. Mean concentration (horizontal bar) and 95% confidence intervals are shown for the four samples.

were obvious between mean metal concentration from the analyses of ten individuals and the individual and mean concentrations from the three other tissue samples.

Study Design

Required Sample Sizes

Based on the mean concentrations and associated measures of variation of various metals examined, we computed the sample sizes required to describe the population mean metal concentrations in fish flesh from Killarney Lake. Since only one fish had a concentration of arsenic in excess of the detection limit, it was not possible to design a sample to determine numbers necessary to reliably measure arsenic concentrations. We believe that further sampling of arsenic need not be conducted in Killarney Lake fishes although no acceptable flesh concentrations for arsenic were located in the literature.

Calculations of required sample sizes to describe population mean concentrations of zinc, copper, cadmium, and mercury in Killarney Lake indicated our pilot sampling provided adequate information and additional sampling will not be required (Table 4). To adequately describe the population mean for lead in brown bullheads and yellow perch, 50 and 26 individuals would be required, respectively.

Maximum allowable flesh consumption

We located in the literature acceptable median concentrations for zinc (Nauen 1983), copper (Nauen 1983), lead (Anonymous 1972), cadmium (Anonymous 1989), and mercury (Anonymous 1989) suitable for human consumption. Mean concentrations of zinc, mercury, and copper and 99% tolerance limits for these metals in Killarney Lake fishes were

Table 4. Means (mg/kg), variances, standard errors (std err) of heavy metals , in fishes from Killarney Lake, Idaho. Also shown are D values that are acceptable differences in metal concentrations from means and required sample size to obtain that difference.

Northern Pike		Zn	Cu	Pb	Cd	Hg
mean	(mg/kg)	3.89	0.31	0.06	0.02	0.05
variance		0.72	0.03	3.12E-03	5.87E-05	1.60E-03
std err		0.27	0.06	0.02	2.42E-03	0.01
D value		1.00	0.50	0.10	0.05	0.08
Sample Size		7	3	4	1	4

Largemouth Bass		Zn	Cu	Pb	Cd	Hg
mean	(mg/kg)	3.54	0.31	0.01	0.02	0.05
variance		0.13	0.01	2.67E-05	4.00E-05	3.09E-03
std err		0.11	0.03	1.63E-03	2.00E-03	0.02
D value		1.00	0.50	0.10	0.05	0.08
Sample Size		3	2	1	2	5

Brown Bullhead		Zn	Cu	Pb	Cd	Hg
mean	(mg/kg)	2.95	0.30	0.18	0.04	0.03
variance		0.32	0.02	0.09	1.16E-04	6.04E-04
std err		0.18	0.05	0.09	3.40E-03	0.01
D value		1.00	0.50	0.10	0.05	0.08
Sample Size		4	2	50	4	2

Black Crappie		Zn	Cu	Pb	Cd	Hg
mean	(mg/kg)	4.46	0.27	0.03	0.03	0.07
variance		1.80	2.38E-03	2.89E-04	4.44E-05	2.21E-03
std err		0.42	0.02	0.01	2.11E-03	0.01
D value		1.00	0.50	0.10	0.05	0.08
Sample Size		11	1	2	2	5

Yellow Perch		Zn	Cu	Pb	Cd	Hg
mean	(mg/kg)	4.79	0.31	0.13	0.03	0.09
variance		2.40	0.02	0.04	1.69E-04	0.01
std err		0.49	0.05	0.07	4.34E-03	0.03
D value		1.00	0.50	0.10	0.05	0.08
Sample Size		16	6	26	2	11

consistently lower than those values considered acceptable upper intake limits by Nauen (1983). Mean concentration of cadmium in all species sampled was lower than the acceptable level of 0.07 mg/kg although 99% tolerance limits exceeded the acceptable limits. Lead concentrations averaged considerably lower than acceptable levels and 99% tolerance limits did not exceed acceptable flesh levels.

DISCUSSION

One question that was not addressed in this study was the possible variations in heavy metals content in fish flesh throughout the year. Our sampling was conducted in the late fall, a time when fewer anglers are fishing on the lateral lakes. Within a few months, however, the ice fishery commences (although it is less popular than the early spring pike and later summer bass fishery (Bruce Rich, Graduate Student, University of Idaho, Personal Communication)). Yellow perch were a species of concern for elevated metals levels in Killarney Lake, and they are probably most commonly harvested through the ice. We have not found any literature that has examined temporal changes in metals in fish flesh and therefore assume that concentrations reported in this report are representative of flesh levels throughout the year.

Variability of metal concentrations among individuals of the same species of fish was surprisingly high (Table 1). For example, concentrations of copper in brown bullheads ranged from 0.16 to 0.56 mg/kg and lead ranged from 0.03 to 1.0 mg/kg. Other species such as yellow perch ranged from 0.01 to 0.05 mg/kg cadmium. One determination of cadmium that was identified as an outlier and not used in the computations was 0.87 mg/kg. Two pike samples analyzed initially suggested cadmium concentrations of 0.16 and 0.15 mg/kg. These too were identified as outliers as three other analyses from the same individuals provided comparable values to other pike (Table 3). This high variability contributed to coefficients of variation that exceeded 100% in lead, cadmium, and mercury. Differences among species are probably related to feeding at different levels of the food chain while differences among individuals of a species may be related to differences in deposition of metals within Killarney Lake. Funk et al. (1973) suggested that the distribution of zinc and cadmium in the sediments was related to the wider inlet to Killarney Lake, thereby causing higher concentrations in the interior of the lake. Other possible differences among individuals may not be real but related to sample contamination during analysis.

We conducted replicate samples from the same fillet and had a second lab run analyses to provide quality assurance and quality control. To assess differences associated with laboratory methods, we found that the means from the original sample of ten individuals generally fell within the confidence intervals for the mean of the three subsamples collected from the same fillet (Figures 10-14; Table 4). This replicate sampling provided a measure of precision associated with our results. Analyses conducted at a second lab also provided generally very comparable results (Table 3).

Mean concentration of heavy metals in fishes from Killarney Lake indicated that concentration of some metals have changed little in the last few years. Rabe and Bauer (1976) reported that mean zinc concentrations in muscle tissue ranged from 3.9 mg/kg in largemouth bass to 13.5 mg/kg in brown bullheads (Rabe and Bauer's data converted to mg/kg wet weight by assuming 80% water content in wet weight samples). Our samples of ten averaged 3.54 mg/kg zinc in largemouth bass while brown bullheads were lower at 2.95 mg/kg. Rabe and Bauer (1976) also reported copper concentrations in muscle tissue ranged from 0.17 mg/kg in yellow perch to 0.44 mg/kg in largemouth bass. Mean concentrations of copper from our sampling was 0.31 mg/kg in both yellow perch and largemouth bass. Cadmium levels in muscle tissues in their study were similar among five species and ranged from 0.06 to 0.22 mg/kg. Cadmium levels in fish fillets from our study ranged from 0.02 to 0.12 mg/kg suggesting a decrease in fish flesh from the 1970's.

Funk et al. (1973) conducted heavy metal analyses on pumpkinseed (*Lepomis gibbosus*), yellow perch, black crappie, brown bullheads, and largemouth bass. Mean zinc levels in fish fillets were 6.6, 11, and 10.8 mg/kg (all converted to mg/kg wet weight) for perch, bullhead, and squawfish, respectively. Mean zinc levels for perch and bullheads from our study were 4.79 and 2.95 mg/kg. Funk et al. (1975) found that mean zinc levels ranged from 2.5-13.5 mg/kg; bullheads contained the highest levels, while bass contained the lowest. The overall mean copper concentration was 0.24 mg/kg for all species as compared to 0.30 mg/kg in this study. Copper levels in four species were similar between these two studies while levels in yellow perch were significantly lower. Funk et al. (1973) also reported cadmium levels averaged 0.47 mg/kg in the five species and were not significantly different among species compared to the 0.05 mg/kg overall species mean in this study. Seventeen years have elapsed between their sampling and ours; nine of those years (1981-1989) were devoid of smelting activities. It is likely that our 1989 data reflects real declines in cadmium content of Coeur d'Alene delta

(Killarney Lake) fish.

Hornig et al. (1988) recently reported on composite samples as part of an EPA water quality monitoring effort in the Coeur d'Alene Basin. Mean levels of cadmium in our study were similar ranging from 0.01 to 0.05 mg/kg compared to 0.007 to 0.031 mg/kg in the EPA study for the same species of fish. Lead and mercury concentrations were generally similar but variable between our study and Hornig et al. (1988). Our mean copper concentrations generally were similar ranging from 0.26 to 0.31 mg/kg compared to Hornig et al.'s data which ranged from 0.14 to 0.39 mg/kg copper.

Mean concentrations describe little of the distribution of heavy metals within various fish populations in Killarney and other lakes. For this reason we computed 99% tolerance limits that provided a clearer description of the population metals levels assuming a normal distribution. Our 99% tolerance limits indicated that we were 99% confident that 99% of the fish populations had concentrations of zinc, copper, lead, and mercury that were less than the action levels. Tolerance limits of cadmium were at or exceeded the action level (0.07 mg/kg) for all species but largemouth bass. We believe that these tolerance limits provide the best available description of metal concentrations in fishes from Killarney Lake.

To assess the potential health effects from the metals in Killarney Lake fishes, we located action limits from the literature. Each of these is listed by the specific metal.

Mercury

Mercury has an action limit of 1.0 mg/kg based on the FDA Compliance Policy Guidelines (Anonymous 1972). Laboratory experiments have shown that predatory fish tend to accumulate about 10-15% more mercury than prey fish. Mercury accumulation through the food chain plus absorption from water causes a basic mercury concentration in predators higher than in prey (Jernelov et al. 1971). Studies have shown that concentrations of mercury are largely dependent on age and/or size but with considerable variability in natural systems (Kelso and Frank 1974). In our study we found that highest mercury concentrations were found in yellow perch and crappie but not pike and bass, the top predators in Killarney Lake. We did find that mercury concentrations increased in yellow perch with both increases in length and weight (Figure 8). Based on this action level, mercury concentrations would limit the fish consumption in Killarney Lake (Table 5). The highest concentration in yellow perch would limit suggested maximum fish consumption to 3.08 kg/week, followed by black crappie (4.4 kg/week) and largemouth bass and northern pike (5.4 kg/week).

Cadmium

The general population is exposed to cadmium principally from food and water. Food normally represents the major source of cadmium exposure and available data indicates that the current intake of cadmium from the diet is 70-245 ug/week (mean = 158 ug/wk) (Anonymous 1989). In

TABLE 5. DIETARY INFORMATION FOR CONSUMPTION OF FISH FROM KILLARNEY LAKE, IDAHO.

METAL	SPECIES	MEAN METAL CONCENTRATION (ug/kg)	MEAN DIETARY INTAKE (ug/week)	MAXIMUM DIETARY INTAKE (ug/week)	SUGGESTED MAXIMUM FISH CONSUMPTION (kg/week)
LEAD	Northern Pike	59.7	1750	3000	20.94
	Largemouth Bass	14	1750	3000	89.29
	Brown Bullhead	183	1750	3000	6.83
	Black Crappie	30	1750	3000	41.67
	Yellow Perch	134	1750	3000	9.33
CADMIUM	Northern Pike	16	154	450	18.50
	Largemouth Bass	18	154	450	16.44
	Brown Bullhead	36	154	450	8.22
	Black Crappie	30	154	450	9.87
	Yellow Perch	30	154	450	9.87
MERCURY	Northern Pike	53	14	300	5.40
	Largemouth Bass	53	14	300	5.40
	Brown Bullhead	34	14	300	8.41
	Black Crappie	65	14	300	4.40
	Yellow Perch	93	14	300	3.08

comparison, the World Health Organization recommends that dietary intake of cadmium be limited to 400-500 ug/week (Murphy et al. 1978). The Joint FAO/WHO Expert Committee (Anonymous 1989) recommended an average intake of 0.007 mg/kg body weight per week. Assuming an average 70 kg person, 490 ug cd/person/week would be the provisional tolerable weekly intake (PTWI). Based on our samples and the mean concentration of cadmium in yellow perch of 32 ug/kg and 158 ug cadmium in the diet and intake water from other sources, an angler could "safely" consume about 9.9 kg/week of perch flesh to remain below this level (Table 5). Higher amounts of fish flesh from other species could be consumed such as 18.5 kg of northern pike not to exceed a suggested maximum weekly intake. These determinations, however, assume a "normal" intake of cadmium from other sources.

Lead

Provisional tolerable weekly intakes are approximately 3 mg/person or 0.04 mg/kg body weight (Anonymous 1972). Based on these levels, a person could consume over 6.83 kg of bullheads per week, the species with the highest lead concentration in Killarney Lake, assuming that fish is the sole source of lead in the diet.

Zinc

No specific action level is available in the United States (Taylor 1989) but levels naturally occurring in other foods exceed the amount in a fillet of Killarney Lake fish. The average daily requirement for zinc has been estimated to be 10-20 mg per person. A survey of Canadian food found that the zinc content of meat, fish and poultry was between 36.6 and 48.8 mg/kg (fresh weight). They indicated that 98% of the average daily intake of zinc comes from the diet (Taylor et al. 1982). The

Canadian Food and Drug Doctorate tolerance limit for zinc is 100 mg/kg (wet weight) (Murphy et al. 1978). Horning et al. (1988) suggested 45 mg/kg as the action level but his source was not clear. Levels in fish flesh from Killarney Lake were considerably lower (mean for all species = 0.084 mg/kg) than levels reported as upper legal limits for food consumption.

Arsenic

The desired level for human consumption is 0 mg/kg but this is unlikely in most situations (Anonymous 1989). There are many regional and ethnic populations consuming large quantities of fish which suggest arsenic intakes of about 0.05 mg/kg bw/day. The only median International legal limit for arsenic was 1.5 mg/kg reported by Nauen (1983) which appears high relative to other limits and unlikely to be a FDA action level. No reported ill effects from these intake levels were located although we were not able to locate any in-depth investigation of arsenic levels in food intake. With the exception of fish, most foods contain less than 0.25 ug/g of arsenic (Anonymous 1989). Levels in fish in Killarney Lake were generally below detection limits.

Copper

Copper is similar to zinc in that health risks to humans from consumption of contaminated fish is minimal; mammals require the metal and are able to excrete surplus levels (Anonymous 1977). Nauen (1983) listed 20 mg/kg as the median International legal limit for fish consumption. Regardless, copper concentrations in Killarney Lake (mean for all species = 0.298 mg/kg) are considerably lower than acceptable levels we found in all of the literature reviewed.

<u>All Metals</u>

Limits to the consumption of fish flesh from Killarney Lake should be based on the intake of mercury in yellow perch and all the other species sampled except brown bullheads (Table 5). Based on our analyses and comparisons with the literature, the recommended intake of fish flesh in Killarney Lake should not exceed 3.08 kg/week. If this limit of fish flesh dietary intake were not exceeded, maximum dietary intakes for all of these metals would not be exceeded.

RECOMMENDATIONS

1. Potentially high metals content in fish from Killarney Lake, Idaho could pose a human health hazard for unrestricted consumption. Anglers should be informed that human consumption of fish flesh from Killarney Lake should not exceed 3.08 kg/week.

2. Sampling of yellow perch and bullheads to analyze for lead in Killarney Lake should be continued. Sample sizes of at least 50 brown bullheads and 26 yellow perch are required to adequately describe the population parameters of lead in these species. Results of our pilot sampling suggest no further sampling for other species is warranted, if our samples collected in the fall are representative of the time when fish are being harvested and consumed. These additional samples should be collected in the spring season to coincide with the time of maximum collection for human consumption.

3. For comparable results, future analyses should be conducted at the laboratories used in this study.

4. Similar sampling of metal concentrations should be conducted in 5-10 years for lead, cadmium and mercury to assess temporal changes in body content.

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