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REGIONAL COPPER-NICKEL STUDY:

FISH TISSUE ANALYSIS

March 3, 1978

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MINNESOTA ENVIRONMENTAL QUALITY BOARD
REGIONAL COPPER-NICKEL STUDY

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INTRODUCTION

As part of a cooperative agreement between the Minnesota Department of Natural Resources (MDNR) and the Minnesota Environmental Quality Board, the MDNR analyzed fish tissues for body burdens of six metals as a functional portion of the Regional Copper-Nickel Study. The primary assignment of this study was the analysis of fish tissues from northeastern Minnesota. The results of these analyses were to provide a regional characterization of the body burdens of metals associated with copper-nickel mining and provide a baseline for future comparison. In addition, tissues were to be archived for future reference and analysis. The principal item of equipment, a Perkin-Elmer Model 603 atomic absorption spectrophotometer equipped with a graphite furance, was put on order in February, 1976, and delivered in August, 1976. Plumbing, wiring, and general set-up were completed in October, 1976, and preliminary analyses were run in November. The first set of fish tissue data was completed in December, and the total year's sample assignment was completed in August, 1977. The following paper documents these analyses.

QUALITY CONTROL

Since most of the data produced in this study depended on measurements near the detection capabilities of the equipment, it was of primary importance to exercise stringent quality control measures. The most probable vector of contamination was the distilled water supply. Groundwater supplied to the lab was initially treated in a water softener, distilled into a glass holding tank and finally run through Barnstead cation removal and ultrapure deionizing columns, respectively. This system produced very pure water with a specific conductance of less than 1.0 micro-ohms approaching ultrapure

conditions. Blank samples of this water were continually tested and were never found to contain measurable levels of any metal under consideration. Standard stock solutions were prepared according to specifications outlined in the Perkin-Elmer manual, Analytical Methods for Atomic Absorption Spectrophotometry, using either the pure metal (Ni, Cu, Zn, Cd) or a salt of the metal [$Pb(NO_3)_2HgCl_2$]. Stock solutions were stored in specially washed 1 liter polyethylene bottles at a pH of 2 or lower (Robertson, 1968; Struempler, 1973).

Final standards were prepared fresh each day by serially diluting stock solutions to desired concentrations. Standards were compared with the Environmental Protection Agency in lab quality control samples. The results of these analyses can be found in Table 1.

All glass and plasticware were carefully washed in Micro Detergent, rinsed in deionized water, soaked in 20 percent HNO_3 usually overnight, and rinsed several times with deionized distilled water. All acid used in the preparation of blanks, stock, and standard solutions was Ultrex brand (Baker) nitric acid from certified lots. All glassware was borosillicate and plasticware, polyethylene. All liquid transfers in the preparation of standards, digestions, and sample injections were done with various sized Eppendorff micro pipets with disposable polyethylene tips. The tips were rinsed three to four times before use in order to avoid contamination (Benjamin and Jenne, 1976), and each tip was used only once. Efforts to wash and reuse pipet tips proved to be unsuccessful. Polyethylene (whirl-pak, 18 oz.) bags and polystyrene weighing boats were used for tissue storage and transfer. To check if there might be a metal leaching problem in each of these materials, a sample of each was filled with

10 percent HNO_3 and allowed to sit overnight. The leachate was then tested for the six metals under consideration and found to be free of contamination. Extreme care was taken to eliminate possible sources of contamination during dissection. All contact surfaces were limited to teflon, polyethylene, or stainless steel. Stainless steel has been found to be safe for use in tissue dissection (Christian and Feldman, 1970). Latex gloves were worn by lab personnel handling fish, allowing for efficient clean-up between dissections. To minimize metal contamination, analysis was performed in a room separated from the main laboratory and supplied with a filtered air source. Glassware used in the preparation of standards was designated solely for that purpose and separated from General Lab Glassware stores. In general, the EPA handbook for Analytical Quality Control in Water and Wastewater Laboratories (1972) was used as a guideline for quality control measures.

In order to check analytical procedure, two samples of fish tissue (muscle and liver), prepared by this lab, were distributed to four other labs for analysis. These labs included the FDA Laboratory, Minneapolis, EPA Water Quality Lab, Duluth; Agronomy Services Lab, St. Paul; and the Research Lab, Department of Soil Sciences, University of Minnesota, St. Paul. The results of these analyses can be found in Table VII. Generally, there appears to be good comparison between the various labs using atomic absorption technique.

TISSUE PREPARATION

Fish

Fish were received from field crews wrapped in polyethylene bags and packed on ice and/or dry ice. Subsamples of tissue were dissected as soon as

possible in order to assure the best estimate of live weight. Muscle tissue was taken from the area above the lateral line directly posterior to the head of the fish. This allowed for a uniform sampling from an area of maximum tissue bulk which was generally free of bones. Uniform sample location appears to be important since recent studies indicate regionalized concentrations of metals even in muscle tissue (Stiefel, 1976). Ideally, a minimum of 10 gms of tissue were dissected out for analysis, duplicate analysis, possible sample splits, and tissue archives. In smaller fish where 10 gms of tissue was not available, the entire fillet from one side was taken. In most cases, all of the liver tissue was extracted. The exceptions were large fish (northern pike) with livers over 30 gms where only part of the liver was taken. Fish which were very small and a few suckers, which appear to decompose more rapidly, had too little liver tissue to work with and were deleted from the collection. Dissected tissue was placed in pre-tared and labeled polystyrene weighing boats and weighed to the nearest one one-thousandth of a gram. Tissue and weighing boat were then placed in 18 ounce polyethylene whirl-pak sample bags and frozen in preparation for freeze-drying. The subsamples of tissue were transferred to the Meat Science Laboratory, University of Minnesota, St. Paul, for freeze-drying. Weighing boats and tissue were placed intact into the mass freeze-dryer, thus minimizing sample handling. When the drying process was complete, the boats were placed in new whirl-pak bags for storage. In preparation for digestion, approximately one half of the dried muscle tissue, and usually all of the liver tissue, was homogenized in an all glass mortar and pestel. The resulting powder was stored in appropriately-labeled whirl-pak bags pending digestion. Through the entire procedure, chain of custody forms were maintained to ensure proper disposition of samples.

Mammals and Invertebrates

In addi[illegible]n to the primary assignment of fish tissue analysis, 16 small mammal (Clethryonomys gapperi) and 38 invertebrate (composite and individual) tissues were dissected and analyzed. These tissues were not freeze-dried, but all other procedures applicable to fish tissues were applied to these tissues. Mammal tissues included liver and muscle from the left hind leg. Invertebrate tissues included clams foot muscle tissue, crayfish tail muscle tissue, leech whole specimen, snail whole body free of shell, and insect whole. Unfortunately, species designations were not afforded these samples.

TISSUE DIGESTION

General Analysis

The assurance of a complete, homogenized digest became one of the most critical points of the study. This was especially important in the case of liver tissue which contains a higher percentage of oils which were difficult to break down. A great deal of literature research and experimentation was required to find a suitable technique. Anderson (1972) in a comparison of wet and dry ashing technique recommends dry ashing over wet ashing of tissue. In experiments conducted in this laboratory, both methods proved to be unreproduceable. Dry ashing especially affected the more volatile metals, while open wet ashing in HNO_3 failed to digest the samples fully. Both methods required more steps than desired, thus increasing contamination vectors. Fats and oils can be properly broken down in wet digestions by using a combination of acids usually containing perchloric, sulfuric, and/or nitric acids (Chernoff, 1975). However, these acid combinations can be

dangerous and special precautions are required for perchloric acid fumes. Since our lab is not equipped with a fume hood suitable for the use of perchloric acid, this method was not considered. Base digestions can be accomplished using a quaternary ammonium hydroxide such as the commercially available Soluene-350 (Packard Instrument Company). Barlow and Khera (1975) reported good solubilization of tissue using Soluene-350. However, preliminary experimentation in this study revealed precipitation problems and inability to keep spiked samples and standards in solution. Recently, pressure digestions, especially those involving teflon-lined digestion bombs and nitric acid, have proved to be a very reproduceable analytical method (Krinitz and Holak, 1974; Davies and Adrian, 1975; Holak, et al., 1972; Pearce, et al., 1976; Sunderman and Wacinski, 1974; Holak, 1974; and Bernas, 1967). Preliminary experiments confirmed these findings, and Parr Digestion Bombs (Model 4547, 25 ml) were selected as the most feasible method of tissue digestion for this project. Not only did this method provide complete digestion, but it was rapid (2-3 hours), required small volumes of expensive ultrex acid and it was applicable to small amounts of tissue. In addition, it required few steps and was applicable to even the most volatile metals, since it is a closed system. Recovery of spiked samples digested in bombs proved to be well within limits of acceptability (Table 2). Manufacturers' recommendations were followed in charging the digestion bombs. Ideally, the conditions of digestion included 0.1 gm dry tissue ($\simeq$ 0.5 gm wet), and 3 ml ultrex HNO_3 at 150°C for 2 hours. In the case of mammal and some invertebrate samples, tissue was not freeze-dried, and approximately 0.5 gm of wet tissue was used. Occasionally, less than 0.1 gm of dry tissue was available for analysis and conditions were changed to accomodate the digestion.

The digestion bomb produced a clear digestate which required no filtering. The digestate was rinsed from the teflon cup into borosilicate glass volumetric tubes and brought up to 10 ml with deionized distilled water. The diluted digestate was then poured into specially washed and rinsed (see Quality Control) 60 ml polyethylene screw cap bottles with a positive seal (Nalgene 2114) and stored at 5°C pending analysis.

Mercury Analysis

Tissues for mercury analysis were not freeze-dried, since experimentation indicates that some organic forms of mercury would be lost through volatilization. A separate digestion applicable to the technique was used for the mercury analysis. Tissue was removed from the frozen specimen in the same region used for the analysis of other metals by taking a plug with a stainless steel corer. This plug was trimmed at either end with a stainless steel scalpel to approximately .6 gms, weighed, and placed in a graduated 50 ml (25 X 200 mm) pyrex folin digestion tube (Pyrex 7900). Five mls of concentrated 4:1 sulfuric-nitric acid were added to each tube which was in turn placed in a hot water bath (80°C) for 2 hours. The digestate was then cooled in an ice bath and 10 ml of saturated (6 percent) potassium permagenate was added to each tube and mixed on a vortex mixer. These were allowed to set in the ice bath until the reaction subsided. Then 5 ml of 20 percent hydroxalamine hydrochloride was added to clear the remaining permangenate and the tubes were again mixed on the vortex mixer. The digestate was brought up to 50 ml with deionized distilled water pending analysis. Just prior to analysis, 5 ml of 20 percent stanous chloride was added to each tube and immediately covered with a rubber septum. These were shaken several times to release the elemental mercury and analyzed. This procedure is a modification of the cold vapor technique devised by Hatch and Ott, 1968 and expanded on by numerous other experimentors.

ANALYSIS

Tissue digests were analyzed for Cu, Ni, Zn, Cd, Pb, and Hg by atomic absorption spectrophotometry. The principal analytical instrument was a Perkin-Elmer Model 603 atomic absorption spectrophotometer equipped with a deuterium backround correction, HGA 2100 graphite furnace, PRS-10 printer-sequencer and a 056 recorder. The specific conditions of analysis for each metal are listed in Table 3. Generally, Cu and Zn were found to be in concentrations applicable to the use of flame spectrophotometry while flameless technique was required for Ni, Cd, Pb. Hg was analyzed by using the standard cold vapor technique. For this purpose, the graphite furnace assembly was modified so that mercury vapor could be swept through the graphite tube via the internal gas flow. Windows were removed from the furnace to enhance the signal. All heat modes on the furnace control box were turned to zero. Drying and charring times were turned to zero while atomizing time was set at the maximum of 30 seconds. In this way, the spectrophotometer could be run just as if the full furnace capabilities were being employed. With the push of one button, proper gas flow, chart recordings, integration, micro-processing, and printout-sequencing were fully coordinated greatly simplifying the analysis. Mercury vapor was swepth through the furnace by installing two 18 gauge hypodermic needles in the internal gas flow line from the furnace control box. These were inserted through the rubber septum covering the digestion tubes, thus blowing the head space and mercury vapor in the tubes into the spectrophotometer light path. Tissue values were compared to blanks and a series of standards prepared in the same manner. Generally, the conditions of analysis for all metals follow Perkin-Elmer recommendations found in the handbook of analysis. Graphite furnace injections were performed using Eppendorff 10 and 20 µℓ pipets.

Standards for all metals were prepared fresh each day by serially diluting stock solutions. Standards and blanks were acidified to match samples.

SPECIAL CONSIDERATIONS

Tissue digests frequently require the development of special technique in order to separate the metal in question and the complex matrix. This problem is compounded by the difficulties inherent to analysis at or near detection capabilities. In the course of this study, these problems were dealt with as they occurred. The solutions to these problems are basically reflected in the final conditions of analysis in Table 3. However, an expanded explanation of the measures employed to overcome certain difficulties could greatly facilitate future analysis. Additionally, different techniques might be employed from the outset to compensate for problems and save valuable time not only in analysis, but in impromptu experimentation and analysis correction.

Complete digestion is of primary importance in overcoming matrix interferences. If complex compounds can be disassociated, the likelihood of interference is decreased. This is dealt with in the digestion section. Bomb digestions have been shown to provide a complete hydrolysis of organic constituents in rat liver (Sunderman and Wacinski, 1974). However, even with this type of digestion, interfering compounds such as salts are common problems.

The most troublesome metal in this study proved to be cadmium. There were several reasons for this: 1) Cd was the least abundant of all metals studied requiring work near detection levels; 2) Cd, outside of Hg, was the most volatile of the metals studied; 3) Cd is one of the most sensitive

metals in AA technique requiring standards of PPB accuracy and reproduceability; and 4) the wavelength (228.8 nm) utilized in the analysis is also subject to broadband absorption by sodium chloride (Pulido, et al., 1966; Schlesinger and Potter, 1974). These problems were overcome by experimenting with charring times and temperatures. The working parameters finally utilized managed to properly separate the cadmium from the matrix, but the time of analysis was greatly lengthened. The use of the deuterium backround corrector was imperative since backround absorption was evident. It is recommended that future Cd analysis include the addition of PO_4, SO_4, or ammonium salts, such as $(NH_4)_2SO_4$, to decrease the volatility of Cd so that charring temperatures can be increased (Giesy and Wiener, 1977). This would reduce the time required for analysis and facilitate Cd-matrix separation. These matrix modifiers should be added to all standards and blanks for proper comparisons.

Special care should be taken in the analysis of nickel, since a narrow band pass is required to eliminate a nearby nonabsorbing wavelength. Experience has shown that any vibration could alter the monochrometer enough to reduce sensitivity.

Previous mention has been made concerning pipet tips as possible vectors of contamination. Another point of concern is variability in pipet delivery and distribution of sample drops in the graphite tube. To eliminate pipet delivery variation, each sample injection was followed by a rinse injection of blank solution. This procedure helps to improve previously unexplained variations in standard replications.

Finally, great care should be taken to ensure a laboratory air supply which is free of water and oil. The system employed in this study included an

oil-free compressor. A delteck oil filter, two cartridge air dryers, and a pur-gas heatless air dryer. This system provided a more than adequate clean air supply.

PRESENTATION OF RESULTS

The results of the tissue analysis are reported as ppm (mg/Kg) metal for dry and wet weight. For reader convenience and reference, live fish length and weight are reported to the left of the same table (Table 4). Results for mammal and invertebrate samples are reported in a similar fashion in Tables 5 and 6.

TISSUE ARCHIVE

Due to lack of funding, samples collected during the 1977 field season were only processed, freeze-dried, and archived for future analysis. These tissues, in addition to the remaining tissue from the 1976 collection, will be stored at the Chemistry Laboratory, Carlos Avery Game Farm, Forest Lake. The archive will be maintained with records until such time that it is deemed feasible to analyze or destroy them. In case any question concerning the analysis should arise, these records will remain open for review.

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Table 1. EPA quality control samples (trace metals), recovery of metal, µg/liter.

METAL	SAMPLE 1		SAMPLE 2		SAMPLE 3	
	ANALYZED VALUE	TRUE VALUE-EPA	ANALYZED VALUE	TRUE VALUE-EPA	ANALYZED VALUE	TRUE VALUE-EPA
Cd	4.8	5.2	--	--	---	---
Cu	19	16	76	72	104	102
Ni	27	26	47	45	147	152
Pb	25	22	--	--	---	---
Zn	12	11	31	30	180	174

Table 2. Recovery of five metals in tissue digests vs. expected concentrations.

METAL	CONCENTRATION (mg/l)		
	EXPECTED CONC.	RECOVERED	% RECOVERED
Cu	.020	.020	100
Zn	.200	.207	104
Cd	.0050	.0049	98
Ni	.100	.089	89
Pb	.050	.045	90

Table 3. Instrument settings.

Perkin-Elmer AAS, Model 603	ZINC	COPPER	CADMIUM	LEAD	NICKEL
Hollow Cathode Lamp	zinc	copper	cadmium	lead	nickel
Current	15 ma	15 ma	4 ma	10 ma	20 ma
Wave Length	214 nm	325 nm	228.8 nm	283.3 nm	232 nm
Slit Opening	.7 nm	.7 nm	.7 nm	.7 nm	.2 nm
Signal	Conc.	Conc.	Conc.	Conc.	Conc.
Mode	Hold	Hold	PK HT	PK HT	PK HT
Recorder	ABS.	ABS.	ABS.	ABS.	ABS.
Integration Time	5 sec.	5 sec.	10 sec.	7 sec.	5 sec.
Expansion	1	1	1	1	1
Background Correction	no	no	yes	yes	yes
HGA-2100 Graphite Furnace & Controller					
Graphite Tube	---	---	non-pyrolized	pyrolized	pyrolyzed
Drying Temp, Time	---	---	110°C, 50 sec.	110°C, 50 sec.	110°C, 60 sec.
Charring Temp, Time	---	---	250°C, 60 sec.	770°C, 40 sec.	1000°C, 50 sec.
Atomizing Temp, Time	---	---	2100°C, 10 sec.	2300°C, 10 sec.	2700°C, 10 sec.
Parge Gas	---	---	Argon	Argon	Argon
Line Pressure, Flow Rate	---	---	40psi, 20 div	40psi, 20 div	40psi, 20 div
Gas Mode	---	---	Interrupt	Interrupt	Interrupt
Recorder	---	---	Auto	Auto	Auto
Flame & Burner Control Box					
Flame Type	C_2H_2/Air	C_2H_2/Air	------	------	------
Oxidant: Line Pressure, Flow	30psi, 70 div.	30psi, 70 div.	------	------	------
Fuel: Line Pressure, Flow	12psi, 30 div.	8psi, 30 div.	------	------	------

KEY TO TABLE 4

Sample No.	Length(cm)	Weight(gm)	Cu	Ni	Zn	Cd	Pb	Hg
Bob Bay-Birch Lake								
F0001 NP-M	35.5	190						
F0001 NP-L	----	---						
Keeley Creek Bay-Birch Lake								
F0019 BC-M								
F0019 BC-L								
F0165 BB-M	--=-	---						
F0165 BB-L	----	---						

Key to Abbreviations

BB - Burbot
BC - Black Crappie
BG - Bluegill
CC - Creekchub
CM - Composite Minnow
CS - Common Shiner
D - Dace
GS - Golden Shiner
HS - Hybrid Sunfish
LMB - Large Mouth Bass
ME - Muskellunge
NP - Northern Pike
RB - Rock Bass
SHR - Short Head Redhorse
TP - Trout Perch
WE - Walleye
WS - White Sucker
YP - Yellow Perch
M - Muscle
L - Liver

METALS IN FISH FROM THE COPPER-NICKEL STUDY AREA, NORTHEAST MINNESOTA.

Sample	Length (cm)	Weight (gms)	Cu Wet	Cu Dry	Ni Wet	Ni Dry	Zn Wet	Zn Dry	Cd Wet	Cd Dry	Pb Wet	Pb Dry	Hg Wet
F0001NP-M	35.5	190	3.80	18.00	.32	1.5	4.54	21.50	.032	.15	.11	.50	.21
F0001NP-L	35.5	190	8.82	33.50	.92	3.5	70.04	266.00	.039	.15	.21	.80	
F0002NP-M	31.8	140	1.75	8.50	.47	2.3	5.26	25.50	.101	.49	.12	.60	.15
F0002NP-L	31.8	140	3.74	15.50	.60	2.5	39.38	163.25	.036	.15	.30	1.10	
F0003WE-M	27.0	130	4.35	19.50	.96	4.3	6.59	29.50	.047	.21	.20	.90	.33
F0003WE-L	27.0	130	4.23	18.28	1.07	4.6	23.74	102.69	.055	.24	.22	.97	
F0004NP-M	36.0	205	11.11	53.00	.84	4.0	10.07	48.00	.107	.51	.19	.90	.14
F0004NP-L	36.0	205	7.57	33.50	.27	1.2	41.47	183.50	.018	.08	.27	1.2	
F0005NP-M	34.7	150	1.11	6.00	.48	2.6	4.16	22.50	.059	.32	.07	.40	.14
F0005NP-L	34.7	150	7.50	35.50	.72	3.4	45.10	213.50	.025	.12	.36	1.70	
F0006BG-M	19.0	160	24.78	105.00	.33	1.4	19.71	83.50	.005	.02	.17	.70	.10
F0006BG-L	19.0	160	233.30	812.50	———		143.98	518.75	.190	.66	———		
F0007BC-M	27.5	250	2.08	9.00	.21	.9	6.25	27.00	.012	.05	.12	.50	.25
F0007BC-L	27.5	250	3.20	16.00	.42	2.1	20.49	102.50	.200	1.00	.20	1.06	
F0008NP-M	50.2	565	4.78	21.00	.30	1.3	7.74	34.00	.005	.02	.09	.40	.25
F0008NP-L	50.2	565	13.71	45.00	1.46	4.8	35.37	116.00	.040	.13	.61	2.00	
F0009RB-M	19.6	85	.52	2.50	.12	.60	5.46	26.50	.008	.04	.10	.50	.30
F0009RB-L	19.6	85	2.56	13.00	.22	1.1	20.63	105.00	.212	1.08	.10	.50	

Sample	Length (cm)	Weight (Gms)	Cu		Cr		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0010WS-M	22.9	90	.44	2.50	.18	1.0	5.31	30.00	.007	.04	.07	.40	.04
F0011WS-M	20.6	75	.60	3.50	.15	.9	4.45	26.00	.024	.14	.10	.60	.05
F0012WS-M	23.7	155	.53	2.50	.23	1.1	4.97	23.50	.019	.09	.08	.40	.11
F0013WS-M	45.4	800	.57	3.00	.10	.5	3.14	16.50	.004	.02	.08	.40	.23
F0013WS-L	45.4	800	4.41	31.50	.69	4.9	17.91	128.00	.063	.45	.13	.90	
F0014NP-M	39.8	285	.47	2.50	.19	1.0	3.69	19.50	.011	.06	.06	.06	.14
F0014NP-L	39.8	285	6.68	28.50	.47	2.0	41.84	178.50	.035	.15	.37	1.60	
F0015BG-M	11.2	25	1.22	2.50	.34	.7	17.83	36.50	.034	.07	.20	.40	<.10
F0016BG-M	20.9	220	.30	1.50	.08	.4	5.99	30.00	.002	.01	.08	.40	.10
F0016BG-L	20.9	220	2.12	8.50	.40	1.6	28.29	113.50	.177	.71	.20	.80	
F0017WS-M	33.8	385	.37	2.00	.30	1.6	3.65	19.75	.024	.13	.08	.40	.08
F0018WS-M	17.1	35	.47	2.50	.26	1.4	5.74	30.50	.028	.15	.08	.40	.08
F0019BC-M	28.4	240	.79	3.43	.27	1.18	5.67	24.51	.016	.069	.20	.88	.44
F0019BC-L	28.4	240	3.21	15.50	.41	2.00	23.71	114.50	.240	1.160	.19	.90	
F0020BC-M	25.8	200	.34	1.47	.31	1.37	5.17	22.55	.011	.049	.09	.39	.38
F0020BC-L	25.8	200	7.74	36.14	.36	1.68	24.06	112.38	.229	1.069	.59	2.77	
F0021RB-M	12.5	30	.22	1.00	.28	1.30	6.51	30.00	.017	.080	.11	.50	.14
F0021RB-L	12.5	30											

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0022BC-M	24.6	160	.67	2.97	.27	1.19	5.11	22.77	.027	.119	.18	.79	.37
F0022BC-L	24.6	160	1.38	7.43	———		16.13	86.63	.254	1.366	.07	.40	
F0023BG-M	18.1	120	.23	1.00	.30	1.30	5.83	25.00	.026	.110	.16	.70	.06
F0023BG-L	18.1	120	2.48	11.50	.30	1.40	21.71	100.50	.132	.610	.17	.80	
F0024BG-M	20.4	190	.35	1.52	.02	.10	5.25	22.73	.028	.121	.07	.30	.13
F0024BG-L	20.4	190	3.08	10.00	.49	1.60	26.62	86.50	.166	.540	.15	.50	
F0025NP-M	36.5	200	.64	2.91	.15	.68	3.00	13.59	.009	.039	.17	.78	.13
F0025NP-L	36.5	200	13.45	49.50	.24	.90	49.05	180.50	.027	.100	.25	.90	
F0026NP-M	62.9	1300	.46	2.00	.28	1.20	3.46	15.00	.007	.030	.12	.50	.42
F0026NP-L	62.9	1300	4.85	16.83	.17	.59	55.50	192.57	.046	.158	.11	.40	
F0027NP-M	70.3	2260	.28	1.46	.26	1.36	3.68	18.95	.025	.127	.10	.54	.61
F0027NP-L	70.3	2260	2.66	17.33	.26	1.68	9.48	62.15	.048	.317	.08	.50	
F0028NP-M	34.5	140	1.11	5.39	.25	1.96	3.43	16.67	.008	.039	.12	.59	.34
F0028NP-L	34.5	140	14.46	57.84	.42	1.66	65.20	260.78	.056	.225	.22	.88	
F0029NP-M	36.3	185	.52	2.47	.27	1.29	3.66	17.33	.013	.059	.17	.79	.16
F0029NP-L	36.3	185	5.31	18.32	.32	1.09	79.64	274.75	.032	.109	.17	.59	
F0030NP-M	37.3	200	.60	2.97	.16	.79	4.54	22.23	.018	.089	.14	.69	.31
F0030NP-L	37.3	200	5.59	24.75	.13	.59	60.12	266.34	.027	.119	.18	.79	

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0031WS-M	41.0	620	1.91	10.50	.33	1.80	2.91	16.00	.007	.040	.11	.60	.09
F0031WS-L	41.0	620	6.04	37.50	.75	4.64	23.58	146.43	.236	1.464	.12	.71	
F0032WS-M	43.3	750	.64	6.44	.30	2.97	1.38	13.86	.012	.119	.09	.89	.08
F0032WS-L	43.3	750	12.73	48.51	.34	1.29	25.34	96.53	.068	.257	.21	.79	
F0033WS-M	28.2	150	.42	2.48	.22	1.29	2.19	12.87	.010	.059	.08	.50	.05
F0034WE-M	36.4	285	<.10	<.49	.43	2.06	4.86	23.04	.021	.098	.15	.69	.39
F0034WE-L	36.4	285	2.36	9.50	.20	.80	19.73	79.50	.089	.360	.20	.80	
F0035WE-M	28.7	120	1.09	5.00	.35	1.60	5.13	23.50	.028	.130	.11	.50	.32
F0035WE-L	28.7	120	—		—		—		—		—		
F0036WE-M	22.4	65	.85	4.50	—		5.22	27.50	.019	.100	.06	.30	.16
F0036WE-L	22.4	65	—		—		—		—		—		
F0037CM(70)	—	—	1.50	7.00	.52	2.40	26.50	123.50	.036	.170	.09	.40	.07
F0038CM(65)	—	—	2.85	12.12	.57	2.42	56.80	241.41	.033	.141	.14	.61	.11
F0039CM(68)	—	—	.98	4.50	.50	2.30	27.07	124.50	.037	.170	.07	.30	.04
F0040CM(105)	—	—	2.19	10.50	.63	3.0	23.96	115.00	.025	.120	.13	.60	.04
F0041WS-M	39.8	580	.29	1.49	.06	.30	3.40	17.33	.020	.109	.12	.59	.05
F0041WS-L	39.8	580	10.21	42.08	.19	.79	33.64	138.61	.312	1.287	.17	.69	
F0042BC-M	26.1	215	.93	4.00	.07	.30	6.60	28.50	.012	.050	.09	.40	.29

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0042BC-L	26.1	215	3.61	16.84	.17	.81	24.80	115.82	.284	1.327	.13	.61	
F0043BC-M	29.4	345	1.28	5.50	.28	1.20	4.66	20.00	.037	.160	.07	.30	.35
F0043BC-L	29.4	345	2.72	10.50	.21	.80	19.43	75.00	.218	.840	.26	1.00	
F0044BC-M	26.0	200	.93	4.04	.12	.51	4.90	4.90	.070	.303	.19	.81	.14
F0044BC-L	26.0	200	5.35	23.53	.31	1.37	21.84	96.08	.270	1.186	.11	.49	
F0045BC-M	25.2	190	2.66	11.86	.12	.52	6.82	30.41	.009	.041	.19	.83	.17
F0045BC-L	25.2	190	2.37	10.42	.12	.52	21.59	94.79	.152	.667	.14	.63	
F0046BC-M	20.1	95	1.43	6.44	.48	2.18	4.72	21.29	.064	.287	.11	.50	.17
F0046BC-L	20.1	95	1.48	6.44	.25	1.09	21.56	93.56	.178	.772	.18	.79	
F0047BC-M	24.5	175	2.40	10.61	.30	1.31	7.08	31.31	.094	.414	.18	.81	.21
F0047BC-L	24.5	175	2.30	10.00	.29	1.30	19.65	85.50	.149	.650	.35	1.50	
F0048WS-M	51.0	1060	.29	1.50	.08	1.78	2.21	11.50	.054	.280	.08	.40	.16
F0048WS-L	51.0	1060	23.54	92.50	.45	.40	38.17	150.00	.738	2.900	.33	1.29	
F0049NR-M	57.6	870	.80	3.50	.09	.40	5.05	22.00	.076	.330	.07	.30	.25
F0049NR-L	57.6	870	9.18	29.00	.28	.90	36.08	114.00	.028	.090	.09	.30	
F0050WS-M	32.0	235	1.76	9.71	.07	.39	4.58	25.24	.021	.117	.16	.87	.11
F0050WS-L	32.0	235	18.43	83.50	.22	1.00	34.77	157.50	.051	.230	.18	.80	
F0051WS-M	34.0	325	.32	1.58	.17	.84	4.21	21.05	.040	.200	.15	.74	.10

Sample	Length (cm)	Weight (gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0051 WS-L	32.0	235	14.31	57.53	.11	.43	35.57	143.01	.059	.237	.21	.86	
F0052 WS-M	42.8	685	.09	.47	.06	.28	4.01	19.81	.006	.028	.17	.85	.14
F0052 WS-L	42.8	685	21.83	75.54	.25	.87	46.97	162.50	.160	.554	.09	.33	
F0053 NP-M	38.4	265	1.11	5.00	.22	1.00	4.89	22.22	.022	.100	.12	.56	.14
F0053 NP-L	38.4	265	4.79	19.79	.15	.63	38.08	157.29	.030	.125	.18	.73	
F0054 WS-M	43.6	755	4.77	24.24	.28	1.41	5.57	28.28	.040	.202	.20	1.01	.06
F0054 WS-L	43.6	755	5.91	18.56	.13	.41	23.47	73.71	.082	.258	.20	.62	
F0055 NP-M	51.3	590	.69	3.43	.04	.20	3.06	15.20	.028	.137	.06	.29	.23
F0055 NP-L	51.3	590	9.36	26.67	<.03	<.10	35.09	100.00	.037	.105	.10	.29	
F0056 NP-M	56.7	880	.21	.97	.13	.58	4.08	18.45	.015	.068	.09	.39	.20
F0056 NP-L	56.7	880	5.72	19.80	.22	.79	37.92	131.19	.037	.129	.40	1.39	
F0057 NP-M	60.5	1190	.50	2.31	.08	.37	5.56	25.46	.024	.111	.10	.46	.29
F0057 NP-L	60.5	1190	10.21	40.31	.26	1.02	48.05	189.80	.028	.112	.18	.71	
F0058 NP-M	44.1	420	1.47	7.47	.07	.34	3.85	19.54	.054	.276	.09	.46	.21
F0058 NP-L	44.1	420	5.16	22.77	.02	.10	38.39	169.31	.034	.149	.11	.50	
F0059 cm(bp)	—	—	1.44	6.50	.44	2.00	26.33	118.50	.084	.380	.13	.60	.10
F0060 cm(bc)	—	—	1.05	5.00	.48	2.30	25.74	122.50	.050	.240	.08	.40	.08
F0061 NP-M	41.4	280	<.11	<.50	.36	1.58	5.44	23.76	.016	.069	.09	.40	.34

Sample	Length (cm)	Weight (Gms)	Cu		Li		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0061NP-L	41.4	280	22.67	70.50	.06	.20	45.98	143.00	.196	.610	.45	1.40	
F0062RB-M	16.6	80	1.01	4.46	.36	1.58	5.74	25.25	.068	.297	.18	.79	.33
F0062RB-L	16.6	80	1.20	5.29	.37	1.65	20.05	88.82	.234	1.035	.13	.59	
F0063BC-M	28.8	305	.11	.50	———		4.70	20.30	.011	.050	.12	.50	.41
F0063BC-L	28.8	305	1.92	8.00	.29	1.20	16.31	68.00	.206	.860	.26	1.10	
F0064ME-M	77.7	3500	1.08	4.46	.26	1.09	3.01	12.38	.046	.188	.10	.40	.64
F0064ME-L	77 7	3500	8.93	22.06	.28	.68	42.87	105.88	.060	.147	.16	.39	
F0065BC-M	24.4	180	.23	.99	.27	1.19	4.41	19.31	.023	.099	.16	.69	.23
F0065BC-L	24.4	180	2.58	12.38	.21	.99	20.67	99.01	.207	.990	.17	.79	
F0066BC-M	26.1	190	.34	1.47	.31	1.37	4.49	19.61	.007	.029	.27	1.18	.34
F0066BC-L	26.1	190	4.90	23.00	.30	1.40	22.49	105.50	.247	1.160	.15	.70	
F0067BC-M	28.3	300	.69	2.94	.28	1.18	4.13	17.65	.002	.010	.09	.39	.46
F0067BC-L	28.3	300	2.86	13.00	.31	1.40	18.83	85.50	.286	1.300	.18	.80	
F0068BC-M	21.8	190	1.13	5.00	.39	1.70	4.31	19.00	.016	.070	.09	.40	.23
F0068BC-L	2 .8	190	———		———		———		———		———		
F0069WS-M	44.2	740	.83	4.00	.06	.30	3.72	18.00	.023	.110	.15	.70	.34
F0069WS-L	44.2	740	35.08	184.50	.13	.70	32.51	171.00	.099	.520	.25	1.30	
F0070NP-M	29.2	90	.49	2.53	.12	.61	4.38	22.73	.027	.141	.10	.51	.23

(concentration in ug/g)

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0070NP-L	29.2	90	10.27	46.20	.20	.89	68.92	310.13	.051	.228	.25	1.14	
F0071NP-M	42.7	305	.43	1.98	.06	.30	3.11	14.36	.004	.020	.11	.50	.27
F0071NP-L	42.7	305	10.73	41.00	.10	.40	55.76	213.00	.055	.210	.18	.70	
F0072WS-M	77.9	165	.20	1.00	.10	.50	3.35	16.50	.008	.040	.14	.70	.08
F0072WS-L	77.9	165	19.23	84.62	.82	3.59	34.96	153.85	.093	.410	.58	2.56	
F0073WS-M	36.3	335	.39	2.00	.16	.80	3.02	15.50	.006	.030	.10	.50	.17
F0073WS-L	36.3	335	30.60	144.87	.19	.90	54.06	262.18	.109	.526	.19	.90	
F0074WS-M	35.7	390	.38	2.00	.13	.70	4.30	22.50	.011	.060	.17	.90	.16
F0074WS-L	35.7	390	9.18	47.00	.35	1.80	23.63	121.00	.074	.380	.14	.70	
F0075WS-M	45.8	990	2.51	12.87	.14	.69	3.58	18.32	.008	.040	.23	1.19	.31
F0075WS-L	45.8	990	15.55	69.80	.24	1.09	37.49	169.32	.137	.614	.20	.89	
F0076WS-M	46.9	990	.74	3.92	.06	.29	3.62	19.12	.013	.069	.13	.69	.19
F0076WS-L	46.9	990	17.72	72.00	.02	1.00	31.29	153.00	.225	1.100	.18	.90	
F0077NP-M	39.0	280	.54	2.53	.09	.40	4.84	22.73	.004	.020	.13	.61	.31
F0077NP-L	39.0	280	8.60	28.71	.12	.40	71.14	237.62	.033	.109	.30	.99	
F0078WS-M	31.5	240	3.79	20.71	.18	1.00	5.08	27.78	.068	.374	.17	.91	.10
F0078WS-L	31.5	240	20.02	101.50	.16	.80	38.76	196.50	.073	.370	.18	.90	
F0079WS-M	44.5	655	.81	4.08	.06	.30	4.14	20.92	.032	.160	.06	.29	.21

(Concentration in ppm)

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0079WS-L	44.5	655	1.18	6.12	.10	.51	34.63	180.10	.294	1.531	.12	.61	
F0080NP-M	53.5	565	0.31	1.52	.02	.10	5.33	26.33	.006	.030	.04	.20	.36
F0080NP-L	53.5	665	32.50	124.47	.03	.11	66.94	256.38	.095	.362	.06	.21	
F0081NP-M	46.2	400	.61	2.88	.10	.48	4.40	20.67	.027	.125	.08	.38	.26
F0081NP-L	46.2	400	12.38	41.58	.27	.89	41.25	138.61	.047	.158	.15	.50	
F0082WS-M	26.8	130	.45	2.48	.07	.40	25.34	138.12	.013	.069	.09	.50	.07
F0082WS-L	26.8	130	15.63	83.17	.33	1.78	4.19	22.28	.167	.891	.15	.79	
F0083WS-M	43.9	620	.85	4.41	.02	.10	3.31	17.16	.013	.069	.08	.39	.20
F0083WS-L	43.9	620	22.22	106.00	.23	1.10	39.31	187.50	.028	1.340	.08	.40	
F0084NP-M	27.8	75	.45	24.49	.02	.11	5.45	25.79	.007	.032	.09	.42	.17
F0084NP-L	27.8	75	7.12	2.11	.21	.71	54.58	187.75	.074	.255	.21	.71	
F0085HS-M	15.9	50	<.11	<.51	.22	1.01	5.52	25.76	.022	.101	.11	.51	.10
F0086BG-M	24.2	215	.33	1.50	.20	.90	4.91	22.50	.024	.110	.07	.30	.25
F0086BG-L	24.2	215	3.24	12.38	.23	.89	22.55	86.14	3.628	13.861	.08	.30	
F0087WS-M	46.8	940	.64	3.06	.04	.20	3.96	18.87	.004	.020	.04	.20	.16
F0087WS-L	46.8	940	21.04	94.06	.31	1.39	43.52	194.55	.372	1.663	.16	.69	
F0088BG-M	24.3	240	.68	3.00	.02	.10	5.90	26.00	.063	.280	.09	.40	.44
F0088BG-L	24.3	240	2.31	10.00	.07	.30	26.86	116.30	1.155	5.000	.08	.40	
F0085HS-M	15.9	50	.92	9.00	.12	1.20	1.19	105.00					

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0089BGM	18.3	85	.30	1.35	.04	.18	649	28.83	.008	.036	.06	.27	.10
F0089BG-L	18.3	85	2.99	14.58	.09	.42	29.03	141.67	.299	1.460	.09	.42	
F0090BG-M	21.0	190	.57	2.50	.02	.10	5.22	23.00	.011	.050	.07	.30	.08
F0090BG-L	21.0	190	1.99	9.04	.07	.32	24.13	109.57	.370	1.681	.14	.64	
F0091BG-M	18.6	90	6.10	27.27	.68	3.03	11.30	50.51	.029	.131	.63	2.83	.11
F0091BGL	18.6	90	3.71	17.50	.61	2.90	30.19	142.50	.347	1.640	.23	1.10	
F0092NP-M	43.5	385	.49	2.45	.10	.49	4.17	21.08	.012	.059	.06	.29	.23
F0092NP-L	43.5	385	10.76	44.86	.22	.93	56.03	233.64	.061	.252	.18	.75	
F0093NP-M	52.3	590	.20	.92	.16	.74	3.55	16.67	.008	.037	.04	.19	.34
F0093NP-L	52.3	590	12.41	45.19	.32	1.15	54.81	199.52	.063	.231	.16	.58	
F0094NP-M	39.2	205	<.10	<.52	.08	.41	5.23	27.32	.004	.021	.10	.52	.21
F0094NP-L	39.2	205	8.89	33.70	.03	.11	46.89	117.72	.040	.152	.09	.33	
F0095WS-M	26.5	125	.66	3.57	.02	.10	3.79	20.41	.004	.020	.13	.71	.15
F0095WS-L	26.5	125	9.52	52.84	.20	1.13	24.77	137.50	.078	.432	.37	2.05	
F0096WS-M	44.0	595	1.02	4.95	.08	.40	5.02	24.26	.072	.347	.10	.50	.27
F0096WS-L	44.0	595	22.33	98.02	.41	1.78	36.09	158.42	.144	.633	.16	.69	
F0097CM(CS)	—	—	1.25	6.38	.21	1.06	82.96	423.94	.123	.628	.12	.64	.14
F0098CM(O)	—	—	3.92	17.00	.16	.70	55.53	241.00	.090	.390	.07	.30	.04

(concentration in mg/kg)

Sample	Length (cm)	Weight (gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0099CM(cc)	—	—	4.39	21.00	.06	.30	34.94	167.00	.107	.510	.15	.70	.07
F0100cm(cs)	—	—	2.02	9.09	.26	1.18	69.35	311.36	.065	.291	.06	.27	.12
F0101 cm (ws)	—	—	2.02	10.11	.23	1.17	21.13	105.85	.064	.319	.08	.43	.05
F0102 cm (ws)	—	—	2.74	13.54	.84	4.17	142.26	704.17	.107	.531	.21	1.04	.15
F0103 WS-M	26.3	140	.83	4.29	.11	.57	5.28	27.14	.013	.067	.07	.38	.11
F0103 WS-L	26.3	140	3.52	18.00	.21	1.09	22.30	114.00	.053	.270	.14	.70	
F0104 cm (ws)	—	—	1.23	6.00	1.17	5.7	24.70	120.75	.020	.10	.06	.30	.07
F0105 cm (cc)	—	—	4.24	19.00	3.39	15.2	25.18	112.75	.031	.14	.07	.30	.05
F0106WE-M	35.5	320	.51	2.38	.13	.60	4.47	20.83	.010	.048	.10	.48	.25
F0106WE-L	35.5	320	3.96	18.45	.25	1.17	18.54	86.41	.238	1.107	.15	.68	
F0107WS-M	30.4	250	.71	2.94	.08	.38	6.77	27.94	.010	.039	.08	.38	.19
F0107WS-L	30.4	250	4.15	17.16	.24	.98	22.91	94.61	.038	.157	.17	.69	
F0108WS-M	29.7	235	.49	2.50	.04	.20	5.08	26.00	.018	.090	.12	.60	.17
F0108WS-L	29.7	235	6.26	34.31	.09	.49	20.66	113.24	.114	.627	.11	.59	
F0109NP-M	210	60	1.00	5.00	.26	1.30	6.18	31.00	.036	.180	.20	1.00	.13
F0109NP-L	210	60	11.88	53.21	.54	2.44	32.05	143.59	.037	.167	.20	.90	
F0110RB-M	16.5	75	.86	3.80	.05	.22	6.13	27.17	.015	.065	.07	.33	.19
F0110RB-L	16.5	75	2.10	8.59	.17	.71	18.32	74.75	.040	.162	.12	.51	

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0111RB-M	14.0	65	.63	2.88	.02	.10	5.84	26.92	.017	.077	.08	.39	.17
F0111RB-L	14.0	65	2.03	8.67	.05	.20	15.14	64.80	.136	582	.17	.71	
F0112NP-M	22.7	50	.30	1.50	.04	.20	5.65	28.00	.012	.060	.08	.40	.08
F0112NP-L	22.7	50	8.98	41.05	.09	.42	44.45	203.16	.032	.147	.14	.63	
F0113NP-M	19.8	50	1.04	4.46	.14	.59	8.44	36.14	.021	.089	.25	1.09	.16
F0113NP-L	19.8	50	—		—		—		—		—		
F0114NP-M	29.5	135	.67	3.47	.11	.59	4.95	25.74	.015	.079	.11	.59	.14
F0114NP-L	29.5	135	5.81	27.00	.09	.40	36.56	170.00	.099	.450	.13	.60	
F0115RB-M	13.8	65	4.61	21.29	.19	.89	7.39	34.16	.019	.089	21	.99	.11
F0115RB-L	13.8	65	1.85	7.43	.22	.89	13.33	53.47	.227	.911	.10	.40	
F0116WS-M	25.7	185	.74	3.92	.04	.20	6.03	31.86	.045	.235	.07	.39	.16
F0116WS-L	25.7	185	7.74	34.62	.52	2.31	25.81	115.38	—		.21	.92	
F0117RB-M	12.5	40	.73	3.40	.19	.87	5.71	26.70	015	.068	.10	.49	.13
F0117RB-L	12.5	40	1.14	5.13	.20	.90	14.45	64.73	.132	.589	.14	.64	
F0118NP-M	37.2	290	60	3.13	.04	.21	5.83	30.21	.020	.104	.16	.83	.13
F0118NP-L	37.2	290	8.98	46.15	.04	.19	28.62	147.12	.034	.173	.06	.29	
F0119NP-M	30.8	150	.20	1.00	.04	.20	4.74	24.00	.008	.040	.10	.50	.06
F0119NP-L	30.8	150	11.73	53.50	.09	.40	43.64	199.00	.099	.450	.18	.80	

(Concentration in mg/kg)

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0120WS-M	30.9	310	.28	1.44	.04	.19	6.27	32.69	.011	.058	.18	.96	.19
F0120WS-L	30.9	310	3.11	14.08	.32	1.46	21.65	98.06	.058	.262	.13	.58	
F0121SHR-M	24.6	165	.76	3.43	.33	1.47	4.69	21.08	.008	.039	.15	.69	.15
F0121SHR-L	24.6	165	4.71	18.81	.37	1.49	15.25	64.85	.117	.465	.07	.30	
F0122SHR-M	21.7	100	1.08	5.00	.24	1.10	7.42	34.50	.071	.330	.07	.30	.06
F0122SHR-L	21.7	100	10.90	14.50	.90	1.20	51.88	69.00	.338	.450	.38	.50	
F0123LM3-M	16.5	80	.98	4.95	.35	1.78	4.52	22.78	.018	.089	.10	.50	.07
F0123LM8-L	16.5	80	2.86	12.50	.34	1.50	18.08	79.00	.080	.350	.14	.60	
F0124cm(CS)	—	—	1.03	4.69	.27	1.25	53.14	288.59	.043	.198	.09	.42	.06
F0125NP-M	38.5	230	.37	1.96	.06	.29	4.59	25.00	.009	.050	.09	.50	.19
F0125NP-L	38.5	230	8.37	37.75	.15	.69	28.80	129.90	.059	.265	.15	.69	
F0126NP-M	32.4	135	.28	1.49	.15	.79	3.87	20.30	.017	.088	.06	.30	.14
F0126NP-L	32.4	135	6.38	32.52	.23	1.17	27.13	138.35	.044	.233	.06	.29	
F0127cm(TP)	—	—	1.01	4.55	.49	2.22	34.41	155.56	.080	.364	.13	.61	.09
F0128cm(CS)	—	—	1.38	5.45	.38	1.49	44.87	177.23	.043	.168	.13	.50	.08
F0129cm(WS)	—	—	6.85	21.72	.80	2.53	31.71	100.51	.073	.232	.29	.91	.07
F0130cm(TP)	—	—	1.60	8.00	.36	1.80	37.17	185.50	.032	.160	.12	.60	.06
F0131cm(WS)	—	—	1.69	8.15	.16	.76	112.99	543.48	.034	.163	.09	.43	.07

Sample	Length (cm)	Weight (gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0132WS-M	51.4	1200	.42	2.45	.05	.294	3.07	18.05	.051	.301	.03	.20	.33
F0132WS-L	51.4	1200	15.68	76.50	.84	4.10	29.30	143.00	.135	.660	.10	.50	
F0133WS-M	21.0	75	1.05	6.31	.13	.78	2.82	16.99	.015	.087	.13	.78	.09
F0133WS-L	21.0	75	20.74	106.00	.21	1.07	26.74	136.67	.039	.200	.10	.53	
F0134WS-M	19.7	40	.65	3.96	.34	2.08	4.23	25.74	.170	1.040	.08	.50	.07
F0134WS-L	19.7	40	—		—		—		—		—		
F0135WS-M	19.4	70	.41	2.50	.36	2.20	3.03	18.50	.018	.110	.13	.80	.08
F0135WS-L	19.4	70	10.83	54.59	.20	1.02	23.59	119.39	.101	.510	.12	.61	
F0136WS-M	21.2	90	.08	.50	.07	.40	3.35	20.00	1.625	9.700	.08	.50	.13
F0136WS-L	21.2	90	14.58	99.26	.09	.59	24.19	164.71	1.058	7.206	.06	.44	
F0137NP-M	39.7	230	.57	3.00	.40	2.10	5.94	31.50	.023	.120	.09	.50	.60
F0137NP-L	39.7	230	18.61	85.80	.39	1.30	29.61	136.50	.102	.470	.11	.50	
F0138NP-M	38.3	190	.98	4.95	.23	1.19	7.14	36.14	.018	.089	.14	.69	.35
F0138NP-L	38.3	190	9.46	49.00	.52	2.70	24.42	126.50	.037	.190	.29	1.50	
F0139NP-M	31.7	150	1.10	5.46	.14	.70	5.00	24.75	.032	.158	.18	.89	.25
F0139NP-L	31.7	150	6.45	28.64	1.73	7.69	35.75	158.74	.037	.165	.07	.29	
F0140NP-M	48.6	400	.29	1.50	.43	2.20	4.58	23.50	.019	.100	.06	.30	.61
F0140NP-L	48.6	400	17.94	82.18	.54	2.47	35.02	160.40	.074	.337	.22	.99	

Sample	Length (cm)	Weight (gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0141WE-M	27.9	150	.31	1.50	.31	1.50	3.92	19.00	.037	.180	.10	.50	.37
F0141WE-L	27.9	150	1.19	5.50	.22	1.00	15.04	69.50	.030	.140	.07	.30	
F0142WE-M	21.0	70	.75	3.50	.70	3.30	4.37	20.50	.038	.180	.17	.80	.13
F0142WE-L	21.0	70	1.32	5.00	.21	0.80	16.05	61.00	.021	.080	.08	.30	
F0143YP-M	24.3	160	1.68	8.00	.29	1.40	5.67	27.00	.013	.060	.15	.70	.12
F0143YP-L	24.3	160	2.32	8.50	.22	.80	22.44	75.00	.074	.270	.08	.30	
F0144YP-M	24.5	135	<.11	<.51	.21	1.02	5.10	24.49	.006	.031	.06	.31	.14
F0144YP-L	24.5	135	4.73	18.50	.31	1.20	23.91	93.50	.107	.420	.13	.50	
F0145WS-M	17.6	50	.93	4.90	.67	3.53	4.18	22.06	.019	.098	.15	.78	.11
F0145WS-L	17.6	50	9.64	48.13	.53	2.63	20.42	101.88	.033	.163	.18	.88	
F0146YP-M	26.8	210	.29	1.47	.21	1.08	3.75	19.12	.006	.029	.04	.20	.12
F0146YP-L	26.8	210	17.56	81.50	.28	1.30	31.47	146.00	.293	1.360	.11	.50	
F0147WS-M	42.9	845	.60	3.40	.17	.97	4.66	26.21	.021	.117	.10	.58	.25
F0147WS-L	42.9	845	12.37	56.80	.15	.68	28.66	131.55	.093	.427	.17	.78	
F0148NP-M	50.0	600	.51	2.45	.24	1.18	3.76	18.14	.006	.029	.12	.59	.51
F0148NP-L	50.0	600	10.38	41.41	.18	.71	29.62	118.18	.159	.636	.08	.30	
F0149NP-M	48.0	490	.66	3.50	.08	.40	4.17	22.00	.006	.030	.15	.80	.32
F0149NP-L	48.0	490	6.83	26.50	<.03	<0.1	21.52	83.50	.052	.200	.10	.40	

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0150NP-M	46.3	440	.38	1.98	.04	.20	3.86	20.30	.013	.069	.09	.50	.32
F0150NP-L	46.3	440	6.27	27.23	.21	.90	28.06	121.78	.057	.248	.18	.80	
F0151NP-M	45.5	460	2.88	14.22	.50	2.45	6.86	33.82	.028	.137	.38	1.80	.25
F0151NP-L	45.5	460	7.39	30.30	.67	2.73	27.35	112.12	.044	.182	.12	.51	
F-0152 WS-M	47.9	945	.65	3.40	.28	1.46	3.53	18.45	.007	.039	.17	.87	.17
F0152WS-L	47.9	945	1.03	4.90	.55	2.65	23.48	112.25	.209	1.000	.37	1.77	
F0153YP-M	46.4	925	.97	5.00	.79	4.10	4.26	22.00	.021	.110	.16	.80	.19
F0153YP-L	46.4	925	16.76	73.08	.51	2.21	28.12	122.60	.130	.567	.20	.87	
F0154YP-M	18.3	65	<.10	<.50	.50	2.48	3.70	18.32	.020	.099	.08	.40	.17
F0154YP-L	18.3	65	5.05	26.50	.17	.90	23.71	124.50	.210	1.100	.10	.50	
F0155YP-M	20.0	85	.11	.50	.45	2.10	4.43	20.50	.030	.140	.13	.60	.18
F0155YP-L	20.0	85	3.51	15.50	.16	.70	22.51	99.50	.281	1.240	.14	.60	
F0156YP-M	17.8	45	.39	2.00	.25	1.30	4.05	21.00	.010	.050	.14	.70	.15
F0156YP-L	17.8	45	17.20	81.00	.25	1.20	29.83	140.50	.327	1.540	.13	.60	
F0157YP-M	17.3	40	.83	4.08	.25	1.22	4.14	20.41	.012	.061	.08	.41	.11
F0157YP-L	17.3	40	34.06	147.47	.07	.30	36.16	156.57	.462	2.000	.07	.30	
F0158YP-M	22.2	100	.20	.98	.57	2.84	3.64	18.14	.197	.980	.06	.29	.28
F0158YP-L	22.2	100	31.23	138.35	.26	1.17	4.27	18.93	.116	.515	.15	.68	

Sample	Length (cm)	Weight (Gms)	Cu		Ni		Zn		Cd		Pb		Hg
			Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
F0159WS-M	33.8	270	.98	5.50	.37	2.10	3.92	22.00	.016	.090	.14	.80	.07
F0159WS-L	33.8	270	20.20	114.14	.95	5.35	25.03	141.41	.105	.596	.18	1.01	
F0160WS-M	26.8	140	.60	3.47	.09	.50	3.80	21.78	.014	.079	.05	.30	.04
F0160WS-L	26.8	140	18.08	87.50	.14	.69	25.54	123.61	.057	.278	.37	1.81	
F0161WS-M	47.4	1040	.55	2.97	.13	.69	3.39	18.32	.007	.040	.11	.59	.17
F0161WS-L	47.4	1040	12.59	68.14	.09	.39	22.74	123.04	.343	1.490	.14	.59	
F0162NP-M	48.0	400	<.10	<.50	.06	.30	4.01	21.00	.006	.030	.12	.60	.30
F0162NP-L	48.0	400	26.18	93.50	.76	2.70	52.24	186.50	.092	.330	.78	2.80	
F0163NP-M	40.2	245	.31	1.50	.10	.50	3.59	17.50	.006	.030	.06	.30	.32
F0163NP-L	40.2	245	12.75	50.98	.17	.69	35.30	141.18	.061	.245	.10	.39	
F0164BB-M	—	—	1.79	9.90	.30	1.68	6.73	35.64	.018	.099	.14	.79	.17
F0164BB-L	—	—	10.06	34.50	.20	.70	22.89	78.50	.017	.059	.09	.30	
F0165BB-M	—	—	.26	1.49	.36	2.08	5.86	33.66	.036	.208	.07	.40	.15
F0165BB-L	—	—	10.44	59.90	.17	.99	19.49	111.88	.152	.871	.17	.99	

Date February 1977

For

EQC Copper-Nickel Regional Impact Study

Sampling Period

12/15/[illegible] through 2/15/77

TABLE I - BODY BURDENS OF SIX METALS IN VOLES (CLETHRIONOMYS GAPPERI) FROM THE COPPER-NICKEL STUDY AREA NORTHEAST MINNESOTA

Collection Station(s) Gabbro and Granite

Collection Date November 11, 1976

Collection Description [illegible] Clethrionomys Gapperi)

Specimen	Tissue	Field Number*	(Conc. in mg/kg) Month Analysis Performed	Cu		Fe		Zn		Cd		Pb		Hg	
Vole	Liver	M/11/17/76/05	June	4.50	Jan	.097	June	57.18	Mar.	0.054	Feb	.24			Gabbro-Spruce
Vole	Muscle	M/11/17/76/05	June	4.25	Jan	.566	June	41.63	Mar.	0.040	Feb	.68			Gabbro-Spruce
Vole	Liver	M/11/17/76/05	June	4.48	Jan	3.251	June	26.35	Mar.	0.052	Feb	.34			Granite-Alder
Vole	Muscle	M/11/17/76/05	June	3.25	Jan	.475	June	32.75	Mar.	0.040	Feb	.36	April	>.02	Granite-Alder
Vole	Liver	M/11/17/76/16	June	6.03	Jan	.670	June	24.90	Mar.	0.054	Feb	.50			Gabbro B.-Spruce
Vole	Muscle	M/11/17/76/16	June	3.91	Jan	.135	June	26.23	Mar.	0.017	Feb	.38			Gabbro B.-Spruce
Vole	Liver	M/11/17/76/36	June	4.15	Jan	.196	June	24.02	Mar.	0.029	Feb	.30			Granite B.-Spruce
Vole	Muscle	M/11/17/76/36	June	7.55	Jan	5.47	June	33.91	Mar.	0.040	Feb	.35			Granite B.-Spruce
Vole	Liver	M/11/18/76/04	June	4.63	Jan	.238	June	27.12	Mar.	0.040	Feb	.24			Gabbro.-Alder
Vole	Muscle	M/11/18/76/04	June	3.71	Jan	.610	June	28.38	Mar.	0.021	Feb	.13	April	> 02	Gabbro-Alder
Vole	Liver	M/11/19/76/04	June	5.14	Jan	.142	June	26.15	Mar.	0.037	Feb	.41			Granite B-Spruce
Vole	Muscle	M/11/19/76/04	June	3.17	Jan	.212	June	27.54	Mar	0.021	Feb	.24			Granite B-Spruce

* Field Number in the case of these mammals is composed of the date of collection plus trap number (last two digits)

TABLE V (cont)

Number 1
Date February 15, 1977
For
EQC Copper-Nickel Regional Impact Study
Covering Period
12/15/76 Through 2/15/77

Collection Station(s) Gabbro and Granite

Collection Date November 11, 1976

Collection Description Mammals (Clethrionomys Gapperi)

Species	Tissue	Field Number	(Conc. in mg/kg) Month Analysis Performed Cu	Ni	Zn	Cd	Pb	Hg	Remarks
Vole	Liver	M/11/19/76/05	5.06	.057	25.19 Mar.	0.038	1.05		Gabbro-Alder
Vole	Muscle	M/11/19/76/05	3.50	.308	25.49 Mar.	0.042	.48		Gabbro-Alder
Vole	Liver	M/11/19/76/17	4.28	.102	23.93 Mar.	0.067	.33		Granite-Alder
Vole	Muscle	M/11/19/76/17	3.12	.382	22.58 Mar.	0.038	.22		Granite-Alder

Date February 15, 1977

For

EQC Copper-Nickel Regional Impact Study

Covering Period

12/15/[illegible] Through 2/15/77

TABLE VI. BODY BURDENS OF 6 METALS IN SELECTED INVERTEBRATES FROM THE COPPER-NICKEL STUDY AREA, NORTHEAST MINNESOTA.

Collection Station(s) Listed in Remarks Column

Collection Date July 1976

Collection Description Invertebrates

Species	Tissue	Field Number	(Conc. in mg/kg) Month Analysis Performed Cu	Ni	Zn	Cd	Pb	Hg	Remarks
*Crayfish	tail	A0023-I	Feb 10.67	Jan .291	Dec 15.31	Mar. 0.029	Feb .08	Mar. 0.14	Partridge R.-1
*Leech	Whole	A0024-I	Dec 2.60	Jan .035	Dec 20.31	Mar. 0.031	Feb .10	Mar 0.07	Birch Lake-2
Clam	foot	A0003-I-1	Dec .50	Jan .446	Dec 8.29	Mar. 0.054	Feb .35	Mar 1.08	Garden Lake-2
Clam	foot	A0003-I-2	Dec .70	Jan .303	Dec 10.35	Mar. 0.079	Feb .40	Mar 1.05	Garden Lake-2
Clam	foot	A0003-I-3	Dec .87	Jan .447	Dec 11.29	Mar. 0.062	Feb .15	Mar 0 05	Garden Lake-2
Clam	foot	A0012-I-1	Dec 2.77	Jan .451	Dec 7.07	Mar. 0.084	Feb .16	Mar 0.05	Birch Lake-2
Clam	foot	A0012-I-2	Dec 2.30	Jan .299	Dec 10.00	Mar. 0.060	Feb .21	Mar 0.06	Birch Lake-2
Clam	foot	A0012-I-3	Dec 1.90	Jan .531	Dec 12.97	Mar. 0.051	Feb .25	Mar 0.08	Birch Lake-2
Clam	foot	A0012-I-4	Dec 2.65	Jan .190	Dec 11.59	Mar. 0.078	Feb .31	Mar 0 13	Bob's Bay Birch Lake-2
Clam	foot	A0012-I-5	Dec 1.93	Jan .214	Dec 10.06	Mar. 0.047	Feb .11	Mar 0.08	Bob's Bay Birch Lake-2
Clam	foot	A0012-I-6	Dec .91	Jan .221	Dec 6.74	Mar. 0.048	Feb .14	Mar 0.06	Bob's Bay Birch Lake-2
Clam	foot	A0014-I-1	Dec .55	Jan .110	Dec 10.82	Mar. 0.047	Feb .11	Mar 0 08	Dunka Bay Birch Lake-2

TABLE VI (CONT)

Date February 15, 1977

For

EqC Copper-Nickel Regional Impact Study

Covering Period

12/15/76 Through 2/15/77

Collection Station(s) Listed in Remarks Column.

Collection Date July 1976

Collection Description Invertebrates

Specimen	Tissue	Field Number	(Conc. in mg/kg) Monthly Analysis Period	Cu	Ni	Zn	Cd	Pb	Hg	Collection Site Remarks
Clam	feet	A0008-I-6	Dec	1.03	.143	8.50	0.086	.02	0.11	Birch Lake-2
*Snail	body	A0009-I	Dec	14.35	1.290	15.48	0.106	1.07	0.09	Birch Lake-2
*Clam	feet	A0011-I	Dec	1.39	.693	9.70	0.072	10.0	0.06	Birch Lake-2
*Crayfish	tail	A0035-I	Dec	5.43	.198	11.48	0.015	.05	0.11	Stoney River-1
*Insect	whole	A0034-I	Dec	4.16	.271	27.23	0.056	.12	0.32	Bear Isl. R.-1
*Crayfish	tail	A0036-I	Dec	9.00	.280	17.27	0.036	.14	0.06	St. Louis R.-1
*Insect	whole	A0032-I	Dec	4.10	1.856	41.82	0.092	.15	0.21	Stoney River-1
*Crayfish	tail	A0029-I	Dec	10.23	.173	22.35	0.041	.14	0.14	Partridge R.-5
*Snail	body	A0028-I	Dec	2.27	1.203	18.05	0.160	.29	0.10	Dunka River-1
*Crayfish	tail	A0027-I	Dec	13.63	.029	15.07	0.020	.09	0.06	Dunka River-1
*Crayfish	tail	A0033-I	Dec	7.01	.714	14.29	0.032	.10	0.15	Embarass R.-1
*Crayfish	tail	A0021-I	Dec	15.50	1.481	23.05	0.034	.12	0.14	Kawishwi R-1

* Composite Sample

TABLE VI (CONT)

January 15, 1977

For

EQC Copper-Nickel Regional Impact Study

Covering Period

12/1/76 Through 2/15/77

Collection Station(s) Listed in Report Column

Collection Date July 1976

Collection Description Invertebrates

Species	Tissue	Field Number	(Conc. in mg/kg) Metal Analysis Performed												Collection Site
			Cu		Ni		Zn	Cd		Pb		Hg			
Clam	foot	A0014-I-2	Dec	1.14	Dec	.076	Dec	9.11	Mar	0.048	Feb	.48	Mar	0.86	Dunka Pit-2
Clam	foot	A0014-I-3	Dec	.64	Dec	.892	Dec	8.33	Mar	0.051	Feb	.51	Mar	0.09	Dunka Pit-2
Clam	foot	A0014-I-4	Dec	.96	Dec	.929	Dec	9.14	Mar	0.058	Feb	.32	Mar	0.09	Dunka Pit-2
Clam	foot	A0014-I-5	Dec	.89	Dec	1.065	Dec	8.58	Mar	0.059	Feb	.41	Mar	0.14	Dunka Pit-2
Clam	foot	A0014-I-6	Dec	.60	Dec	.995	Dec	6.22	Mar	0.045	Feb	.24	Mar	007	Dunka Pit-2
*Clam	foot	A0025-I-1	Dec	.59	Dec	.414	Dec	8.88	Mar	0.059	Feb	1.48	Mar	0.40	Dunka Pit-2
*Clam	foot	A0025-I-2	Dec	.24	Dec	.889	Dec	13.70	Mar	0.096	Feb	.44	Mar	0.24	Dunka Pit-2
*Clam	foot	A0025-I-3	Dec	1.10	Dec	.219	Dec	5.90	Mar	0.047	Feb	.28	Mar	0.24	Dunka Pit-2
*Leech	whole	A0026-I	Dec	3.27	Dec	.959	Dec	115.24	Mar	0.029	Feb	.25	Mar	0.21	Dunka Pit-2
Clam	foot	A0008-I-1	Dec	.751	Dec	.064	Dec	9.33	Mar	0.058	Feb	.17	Mar	0.04	Birch Lake-2
Clam	foot	A0008-I-2	Dec	.541	Dec	.35	Dec	9.59	Mar	0.059	Feb	.16	Mar	0.06	Birch Lake-2
Clam	foot	A0008-I-3	Dec	1.14	Dec	.097	Dec	12.01	Mar	0.065	Feb	.36	Mar	7.01	Birch Lake-2

TABLE VI (cont)

TISSUE ANALYSIS PROGRESS REPORT
Numbe
Date February 15, 1977
For
EQC Copper-Nickel Regional Impact Study
Covering Period
12/15/76 Through 2/15/77

Collection Station(s) Listed in Remarks Column

Collection Date July 1976

Collection Description Invertebrates

Species	Tissue	Field Number	(Conc. in mg/kg) Metals Analysis Performed														Collection Site Remarks
				Cu		Ni		Zn		Cd		Pb		Hg			
Clam	foot	A0008-I-4	Dec	.611	Jan	.147	Dec	9.5		0.059	Feb	.27		0.11			Birch Lake-2
Clam	foot	A0008-I-5	Dec	.624	Jan	.208	Dec	11.35		0.079	Feb	.06		0.15			Birch Lake-2

Table 7. Quality assurance sample split. Comparison of analytical results from five cooperating labs for two tissues.

LAB	INSTRUMENT	Cu	Ni	Zn	Pb	Cd
Muscle ug/g Dried Tissue						
A	A.A.S.	0.7	0.16	15.0	---	.016
B	Induced plasma arc.	8.5	10	28.6	10	1
C	Polarograph A.A.S.	---	---	16.5	0	0
D*	A.A.S.	1.05	1.30	18.4	.31	.15
E	A.A.S.	1.04	---	17.9	.34	.14
Liver ug/g Dried Tissue						
A		13.3	.35	85.9	---	.29
B		27.0	10	73.4	10	1
C		---	---	83.8	.40	.15
D		14.8	1.68	63.9	.39	.32
E		13.12	---	60.6	.34	.21

*Ecological Services Chemistry Lab