2003 and 2004 Walleye and Muskellunge Total Mercury Analyses

by

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INTRODUCTION

Walleye (*Stizostedion vitreum*) and muskellunge (*Esox masquinongy*) are targeted for harvest by Chippewa tribal members from many off-reservation inland lakes in Wisconsin each spring (Krueger 2004). Tribal representatives have expressed concern about the health risk that mercury in fish poses to tribal members. As a result of this concern, the Great Lakes Indian Fish and Wildlife Commission (GLIFWC) has been collecting walleye annually since 1989 during spring from various lakes routinely harvested by tribal members. Muskellunge are collected occasionally. Several funding sources have been used for collection and analysis of the fish for total mercury concentration. The fish were measured for total mercury as a surrogate for methylmercury because most mercury (>95%) in top predator fish is in the form of methyl mercury (Bloom 1992, Lasorsa and Allen-Gil 1995). The data are used to prepare tribal and lake specific GIS maps (Appendix 1). These maps are updated every 2-3 years and made available to tribal members at offices where permits for off-reservation spearing are issued, tribal health centers, schools, libraries, and at public meetings (GLIFWC 2004). The maps were updated in 2005 using a methodology described in Madsen and DeWeese (in prep.).

This report presents results of mercury testing of walleye collected from off-reservation lakes during 2003 and 2004 and of muskellunge collected in 2003. Additional total mercury data collected from muskellunge in 2002 that were not previously reported (Groetsch 2002), and total mercury data from northern pike (*Esox lucius*) collected in 2004 as part of a cooperative project with the University of Wisconsin-La Crosse (UW-La Crosse) are also reported here. Funding for the collection and analysis of these samples is described in Table 1.

Table 1. Summary of funding sources for total mercury analysis (A) and collection (C) of walleye (*Stizostedion vitreum*), muskellunge (*Esox masquinongy*), and northern pike (*Esox lucius*) from ceded territory inland lakes during 2002-2004.

Funded By:	**Walleye**		**Muskellunge**		**Northern Pike**
	2003	2004	2002	2003	2004
EPA[1] Supplemental		A			
EPA STAR[2]		A, C			
ATSDR[3]	A, C		C	A, C	
UW-LAX[4]		A, C			A, C
BIA[5]		C			
WI DNR[6]			A		

[1] United States Environmental Protection Agency
[2] Science to Achieve Results
[3] Agency for Toxic Substances and Disease Registry
[4] University of Wisconsin La Crosse, Mercury Laboratory
[5] Bureau of Indian Affairs
[6] Wisconsin Department of Natural Resources

METHODS

Collection of Samples

Walleye from inland lakes were collected during spring primarily from tribal spearers and netters and Great Lakes Indian Fish and Wildlife Commission (GLIFWC) fishery assessment crews. Some fish were also collected by Mole Lake Band of Lake Superior Chippewa (MLK), Wisconsin Department of Natural Resources (WI DNR), and Minnesota Department of Natural Resources (MN DNR) fisheries assessment crews. Plans called for twelve walleye to be collected with three fish taken from each of four size ranges (12.0 to 14.9, 15.0 to 17.9, 18.0 to 22.0, and greater than 22.0 inches). However, in five lakes where walleye were collected for the UW-La Crosse, plans were to collect twenty fish, five from each of the four size ranges.

Muskellunge from inland lakes were collected during spring 2002 and 2003 from tribal spearers within two size ranges: greater than 35 and less than 35 inches. Northern pike were collected for UW-La Crosse from three lakes by tribal netters and GLIFWC fisheries staff during spring 2004. Plans called for twenty northern pike to be collected, with five fish to be taken within each of four size ranges (20.0 to 24.9, 25.0 to 29.9, 30.0 to 35.0, and greater than 35 inches).

Upon collection, walleye, muskellunge, and northern pike were measured for total length and sex determined. A metal identification tag with a unique number was attached to each fish. Fish were then placed on ice in a cooler and transferred to a freezer (at temperatures at or below -10 °C) within 36 hours. A chain-of-custody form was filled out to identify fish collected from individual lakes each night (Appendix 2). The form also served as a record of who collected and transported the samples and when they were placed on ice or transferred to a freezer. A second chain-of-custody form was used when transferring fish to the Lake Superior Research Institute (LSRI) in Superior, Wisconsin or to the UW-La Crosse Mercury Laboratory in La Crosse, Wisconsin for processing and mercury analysis.

Processing

Walleye were processed into skin-off fillets at GLIFWC (except for those fish processed and analyzed by UW-La Crosse in 2004) using stainless steel knives and cutting surfaces. All surfaces and equipment were washed with a mild dish detergent then rinsed with tap water prior to processing each fish. The following descriptive data were collected from each fish: a second length measurement (denoted as frozen length), sex, round weight, fillet weight, and the second or third dorsal spine was removed for aging. An otolith was also removed from walleye from two lakes in 2004 to use in comparing aging methods (spine vs. otolith). A single skin-off fillet was removed from each walleye, weighed on a digital scale, and placed into a one-gallon plastic bag with an interlocking seal. A water proof sample label containing the name of the lake, fish identification number, year, purpose of collection, species, type of sample and title of study was placed into each bag with the fillet (Figure 1). The tag identification number was recorded on the outside of each bag. All descriptive data were recorded on a laboratory data sheet. All

individually bagged fillets for a given lake were placed into a single 15-gallon plastic bag, sealed, and labeled with the name of the lake. Spines or otoliths were placed into small envelopes with a label, similar to the fillet labels (Figure 1), affixed to the outside of the envelope. The age of the fish was determined by counting the number of annuli (translucent zones) in the spine cross-section consistent with Schram (1989) or in the otolith (Milroy, pers. comm.). Experienced GLIFWC Inland Fisheries technicians aged the spines and otoliths. Age data were entered into GLIFWC's mercury database but are not included as part of this report.

All chain-of custody forms and GLIFWC laboratory data sheets were filed and kept in a three-ring binder at GLIFWC's main office.

Figure 1. Example of a sample label placed into one-gallon walleye fillet bags.

Project: Spring Walleye Sampling For Mercury-2004	Client: GLIFWC
Fish Name: Walleye	Tag No. 0551
Month/Day Collected: 4/23	Year: 2004
Tissue type: Fillet	Sample Processing: Hg
Lake Name: Rest Lake (Vilas)	Processor: LSRI

Total Mercury Analyses

Walleye, muskellunge, and northern pike fillets were received by LSRI, WI DNR, and UW-La Crosse in good condition.

A complete description of fillet grinding, total mercury analysis and associated quality control and assurance is provided in the LSRI laboratory reports (Appendix 7 & 8). Briefly, the fillets were partially thawed and ground three times with a stainless steel motorized meat grinder. An aliquot (200 mg) of the ground tissue was digested and analyzed for total mercury using a Cold Vapor Atomic Absorption Spectroscopy (Perkin Elmer FIMS-100 Flow Injection Mercury Analysis System) method based on EPA Method 245.6. The WI State Laboratory of Hygiene used EPA Method 1631 and Cold Vapor Atomic Absorption Spectroscopy. The UW-La Crosse Mercury Laboratory ground and prepared skin-off walleye and northern pike samples according to their lab protocol. Total mercury analysis at UW-La Crosse was based on EPA Method 1631 using a Leeman Labs Hydra Gold Atomic Fluorescence Spectrophotometer.

Quality Control

Quality control at LSRI was monitored through four methods: 1) the analysis of a certified reference tissue (DORM-2, *Squalus acanthias*) to determine accuracy, 2) tissue spikes to test the extraction method for efficiency and interferences, 3) duplicate analyses to determine precision, and 4) procedural blanks to determine whether sample processing changed the mercury content of the samples.

Data received from WI DNR and UW-La Crosse were verified for quality control by the individual labs. A summary of inter-lab comparison samples analyzed by both UW-La Crosse and LSRI is included in Appendix 3.

Quality assurance reports from audits of both the field collection of samples and laboratory processing and analysis are included in Appendices 4 and 5.

RESULTS

Quality Control

Standard Reference Material

The DORM-2 reference tissue has a certified concentration of 4.64 ± 0.26 µg Hg/g tissue. It was analyzed in duplicate with each batch of 20 samples. The measured reference tissue mean concentration for the 2003 analyses was 4.39 ± 0.32 µg Hg/g tissue (94.6 ± 6.67 % of certified value). The measured mean for the 2004 STAR grant funded analyses was 4.33 ± 0.25 µg Hg/g tissue (93.3 ± 5.33 % of certified value), 4.28 ± 0.33 µg Hg/g tissue (92.3 ± 7.11 % of certified value) for the EPA Supplemental funded analyses, and 4.32 ± 0.39 µg Hg/g tissue (93.2 ± 8.32 % of certified value) for samples analyzed as an inter-lab comparison with UW-La Crosse. These results are within the certified total mercury concentration range and represent good accuracy.

Spikes

Mean percent recovery of tissues spiked with a known amount of mercury was 84.4 ± 13.6 %, 99.8 ± 8.19 %, and 92.4 ± 10.4 % for 2003, 2004 STAR, and 2004 EPA Supplemental, respectively. All of these spike analyses were within LSRI acceptance ranges, which signifies a consistent and efficient extraction method. Two of the tissue spike duplicates (2004) measured as part of the inter-lab comparison with UW-La Crosse had a mean recovery of 95.0 ± 1.78 percent, however, the third spike sample had mean recoveries of 115.5 and 140.8 percent on subsequent analyses and was determined to have interferences. Results from this sample should be interpreted with caution.

Duplicates

Duplicate analyses of 10 % of the samples were 93.1 ± 4.79 %, 92.8 ± 4.60 %, 93.5 ± 5.56 %, and 93.5 ± 2.99 % similar for 2003, STAR, EPA Supplemental, and UW-La Crosse comparison samples respectively. These results were within LSRI acceptance ranges, which suggests good precision.

Procedural Blanks

Procedural tissue blanks (canned tuna, *Thunnus* sp.) were split into two aliquots on each processing day. One aliquot was processed in the same manner as the walleye fillets and the second aliquot was directly digested without processing. The procedural blanks were 87.1 ± 12.2%, 94.1 %, 85.2 ± 11.0%, and 79.0 ± 12.9 % similar for the 2003, STAR, EPA

Supplemental, and UW-La Crosse comparison samples, respectively. Two of the procedural blank samples from 2004 were outside LSRI acceptance ranges (61.2% and 59.4% agreement) and were re-analyzed . These samples were within acceptance limits after re-analysis (78.7% and 88.4% agreement). The concentrations of mercury measured in the tuna blanks is very low compared to other samples, thus a small difference between before and after grinding concentrations will be reflected as a greater percent difference. Overall, the procedural blank percent agreement analyses suggest that processing did not change the mercury content of the samples.

In summary, the quality control data was found to be in good agreement with the quality assurance parameters which demonstrates that the data are precise and accurate.

Sample Results

2002

Fifteen muskellunge collected from 7 lakes in Wisconsin by GLIFWC in 2002 were analyzed by the WI State Laboratory of Hygiene. The muskellunge ranged in length from 31.0 to 44.7 inches. Total mercury concentrations ranged from 0.086 to 1.1 μg Hg/g (Appendix 6).

2003

In 2003, 165 walleye from 15 lakes and 17 muskellunge from 8 lakes in Wisconsin were analyzed for total mercury concentration (Appendix 7). The walleye ranged in length from 11.0 to 28.1 inches. Total mercury concentrations on a wet weight basis in walleye muscle tissue ranged from 0.079 to 1.61 μg Hg/g (parts per million). In addition, 4 walleye from 1 Michigan lake were analyzed. Total mercury concentrations on a wet weight basis ranged from 0.955 to 1.42 μg Hg/g (parts per million) in muscle tissue. Muskellunge ranged in length from 34.0 to 47.5 inches. Total mercury concentrations on a wet weight basis in muskellunge muscle tissue ranged from 0.200 to 1.12 μg Hg/g.

2004

In 2004, 392, 12, and 153 skin-off walleye fillets were collected and analyzed for total mercury from 36 Wisconsin, 1 Michigan, and 13 Minnesota lakes, (Appendices 8 & 9). In addition, 10 walleye collected in 2003 by WI DNR from Squaw Lake, Vilas Co. were analyzed. These walleye had remained frozen since collection and were processed and analyzed along with the 2004 fish.

A total of 17 skin-off northern pike were collected from one Wisconsin lake (Franklin Lake, Forest Co.) and 20 skin-off northern pike fillets were collected from one Minnesota lake (Lake Mille Lacs, Aitkin Co.) (Appendix 9).

Walleye length and mercury data are summarized for each lake in each state in Table 2 (Wisconsin), Table 3 (Michigan), and Table 4 (Minnesota). Briefly, walleye lengths ranged from 12.0 to 27.8 inches from Wisconsin lakes, 13.2 to 23.7 inches from the Michigan lake, and 12.2 to 29.8 inches from Minnesota lakes. Total mercury concentrations on a wet weight basis ranged from 0.089 to 1.20 µg Hg/g from Wisconsin lakes, 0.199 to 1.01 µg Hg/g from the Michigan lake, and 0.054 to 1.26 µg Hg/g from Minnesota lakes.

Table 2. Summary statistics for mercury concentration (ug Hg/g) and fresh length (inches) for walleye collected from Wisconsin lakes during spring 2004.

LAKE	# of Fish	Mean Conc.	Std. Dev. Conc.	Median Conc.	Max. Conc.	Min. Conc.	Mean Length	Std. Dev. Length
ALDER L	8	0.365	0.094	0.401	0.497	0.209	16.7*	3.5
BALLARD L	1	0.683	-	0.683	0.683	0.683	15.2	-
BALSAM L	3	0.171	0.017	0.181	0.181	0.152	17.1	2.2
BASS-PATTERSON L	10	0.298	0.102	0.268	0.510	0.153	17.5	3.6
BEARSKIN L	19	0.195	0.080	0.172	0.375	0.0720	16.3	2.5
BIG PORTAGE L	7	0.302	0.165	0.263	0.661	0.156	15.7	1.6
CLEAR L	6	0.204	0.068	0.192	0.318	0.143	16.3*	2.7
CRAB L	9	0.591	0.115	0.564	0.763	0.369	16.6	3.4
FRANKLIN L	12	0.179	0.132	0.123	0.470	0.0690	18.6	4.5
HIGH L	12	0.374	0.275	0.253	0.931	0.127	18.0	4.3
ISLAND L	10	0.416	0.098	0.419	0.589	0.290	17.0	3.3
L CHIPPEWA	12	0.444	0.203	0.372	0.909	0.174	18.3	4.0
L LAURA	12	0.419	0.155	0.353	0.684	0.257	19.0	4.2
L LUCERNE	12	0.441	0.262	0.367	1.090	0.158	17.9	3.8
L NEBAGAMON	12	0.747	0.229	0.711	1.18	0.452	18.5	3.6
L NOKOMIS	10	0.403	0.179	0.392	0.837	0.176	17.6	3.8
LAC VIEUX DESERT	11	0.201	0.121	0.157	0.468	0.0930	18.2	4.7
LITTLE STAR L	12	0.454	0.188	0.415	0.783	0.255	17.1	2.9
LONG L	12	0.350	0.101	0.375	0.494	0.212	16.8*	2.6
MANITOWISH L	12	0.350	0.174	0.302	0.656	0.157	16.2	2.4
MIDDLE EAU CLAIRE L	12	0.523	0.298	0.417	1.13	0.176	18.1	3.9
NELSON L	8	0.399	0.106	0.399	0.521	0.209	18.1	2.5
PELICAN L	12	0.234	0.081	0.249	0.374	0.116	17.5*	3.1
REST L	8	0.254	0.127	0.217	0.520	0.136	16.1	3.1
ROBERTS L	10	0.395	0.119	0.386	0.617	0.218	17.0	3.5
ROUND L	11	0.220	0.111	0.240	0.470	0.0890	17.4	3.3
SEVENMILE L	13	0.767	0.184	0.776	1.05	0.482	18.1	3.3
SHERMAN L	12	0.409	0.201	0.354	0.740	0.220	18.0	4.2
SPIDER L	10	0.479	0.264	0.358	1.11	0.131	16.1	3.6
SQUAW L	22	0.513	0.147	0.509	0.745	0.215	14.6	1.9
TOMAHAWK L CHAIN	20	0.360	0.176	0.318	0.914	0.206	17.5	3.2
TURTLE-FLAMBEAU FL	7	0.692	0.268	0.610	1.20	0.388	16.0	2.6
UPPER EAU CLAIRE L	12	0.520	0.311	0.422	1.17	0.265	19.0	4.8
UPPER ST CROIX L	10	0.459	0.243	0.462	0.893	0.167	17.9	3.8
WILD RICE L	11	0.344	0.155	0.322	0.566	0.0920	14.3	1.4

* Reported mean includes some fish measured as "frozen length" at GLIFWC laboratory.

Table 3. Summary statistics for mercury concentration (ug Hg/g) and fresh length (inches) for walleye collected from Michigan lakes during spring 2004.

LAKE	#of Fish	Mean Conc.	Std. Dev. Conc.	Median Conc.	Max. Conc.	Min. Conc.	Mean Length	Std. Dev. Length
L GOGEBIC	12	0.437	0.244	0.342	1.01	0.199	18.5	3.6

Table 4. Summary statistics for mercury concentration (ug Hg/g) and fresh length (inches) for walleye collected from Minnesota lakes during spring 2004.

LAKE	# of Fish	Mean Conc.	Std. Dev. Conc.	Median Conc.	Max. Conc.	Min. Conc.	Mean Length	Std. Dev. Length
CHISAGO L	12	0.350	0.228	0.225	0.742	0.110	18.9	5.9
GREEN L	12	0.376	0.213	0.318	0.917	0.165	19.0	5.6
KNIFE L	12	0.181	0.120	0.126	0.486	0.0990	16.1	5.0
LITTLE ELK L	7	0.234	0.131	0.208	0.487	0.108	23.0	2.8
LITTLE ROCK L	7	0.116	0.054	0.100	0.233	0.0760	13.5*	2.3
MILLE LACS L	20	0.188	0.0978	0.144	0.435	0.0872	18.8	3.6
RUSH L (EAST)	12	0.184	0.103	0.165	0.462	0.0760	19.3	5.4
RUSH L (WEST)	12	0.277	0.221	0.233	0.775	0.0540	19.7	5.8
SOUTH BIG PINE L	7	0.659	0.382	0.508	1.26	0.329	16.7	4.2
SOUTH CENTER L	13	0.282	0.152	0.220	0.632	0.135	18.2	4.7
SOUTH LINDSTROM L	12	0.293	0.141	0.259	0.615	0.147	18.9	4.8
SOUTH LONG L	15	0.272	0.140	0.197	0.612	0.141	18.1	3.9
TYPO L	12	0.177	0.057	0.166	0.290	0.113	15.1	1.2

* Reported mean includes some fish measured as "frozen length" at GLIFWC laboratory.

Northern pike lengths ranged from 21.1 to 39.0 inches and total mercury concentrations on a wet weight basis ranged from 0.075 to 0.596 µg Hg/g from the Minnesota lake. Northern pike lengths ranged from 13.0 to 28.9 inches from the Wisconsin lake. Total mercury concentrations in northern pike from the Wisconsin lake ranged from 0.048 to 0.336 µg Hg/g (Appendix 9).

SUMMARY

Walleye total mercury results from 2003 and 2004 and muskellunge results from 2003 were described and are summarized in this report. In addition, previously unreported muskellunge total mercury data from 2002 and northern pike data collected in 2004 as part of a cooperative study with UW-La Crosse were reported. Quality control results determined that the measured total mercury concentrations are precise and accurate. Total mercury concentrations in walleye tended to vary within a lake by size (larger fish generally having higher mercury concentrations) and between lakes for similar size groups of fish. These data have been entered into GLIFWC's mercury database (Madsen 2005) used to produce GIS-based mercury in walleye consumption advisory maps (Madsen and DeWeese, in prep.).

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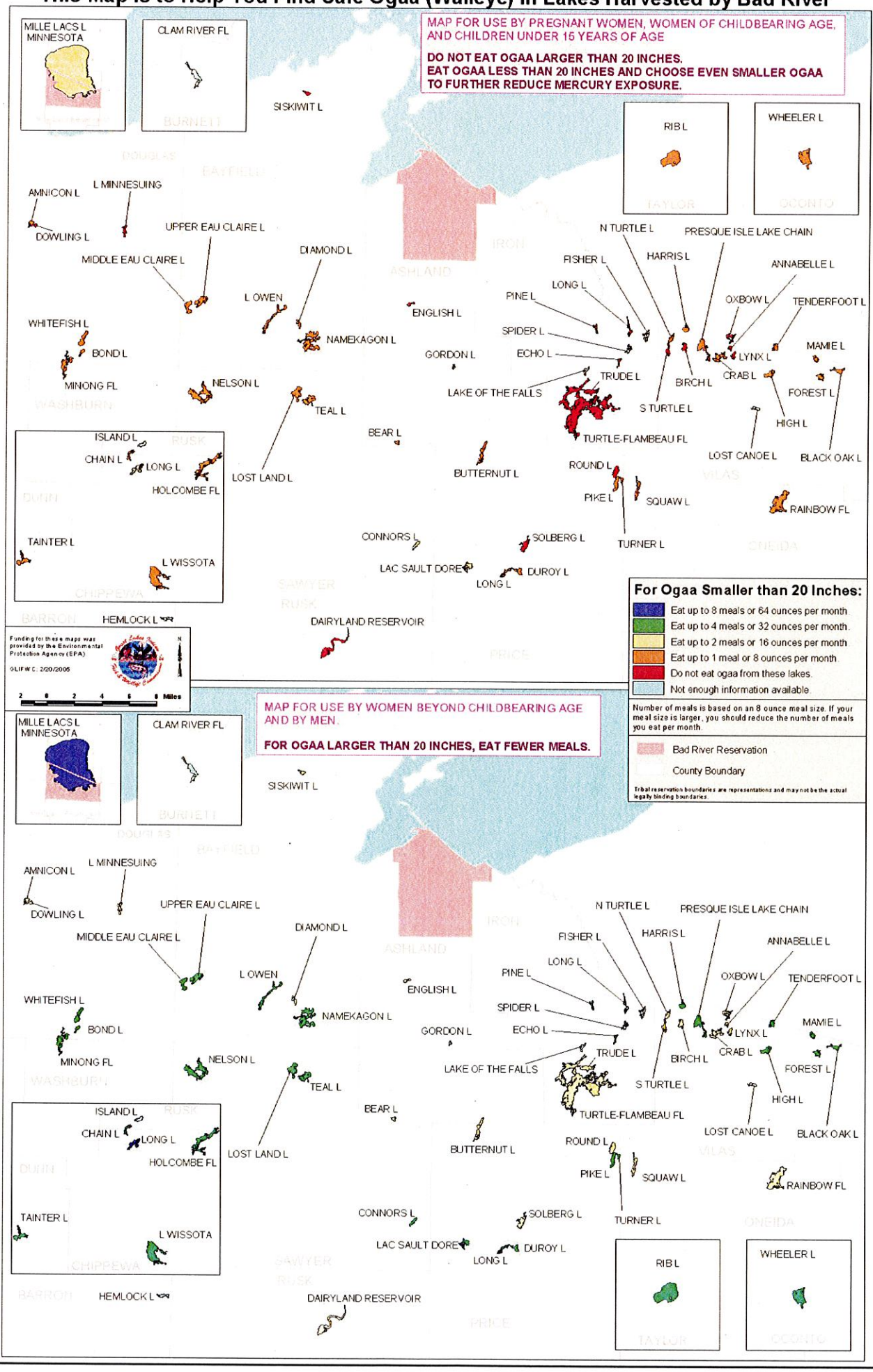
LIST OF APPENDICES

Appendix 1

Example Great Lakes Indian Fish and Wildlife Commission (GLIFWC) Geographic Information System (GIS) - Based Mercury in Walleye Consumption Advisory Map

This Map is to Help You Find Safe Ogaa (Walleye) in Lakes Harvested by Bad River



Recommended Maximum Number of Ogaa Meals per Month For Lakes Harvested by Bad River

SORTING AND LABELING *OGAA* PRIOR TO FREEZING

When Cleaning *Ogaa*:

- Put *ogaa* under 20 inches in bags labeled "under 20 inches."
- Put *ogaa* over 20 inches in bags labeled "over 20 inches".
- Label bags with the lake name.
- Follow the advice below for maximum number of meals per month.

USING THIS CHART TO FIND SAFER *GIIGOONH*

MAXIMUM NUMBER OF MEALS PER MONTH

Advice is for all lakes combined. For example, if you eat four meals in a month from green lakes you should not eat any other meals of *ogaa* in that month.

MEAL SIZE

Meal size is based on 8 ounces. An average 19 inch *ogaa* will have 8 ounces of meat. If your meal size is larger you should eat fewer meals of *ogaa*. If it is smaller you can eat more meals of *ogaa*.

OTHER *GIIGOONH*

Giigoonh such as muskellunge, largemouth bass, smallmouth bass, and northern pike will have more mercury than *giigoonh* such as lake whitefish, herring, bluegill, sunfish, crappie or perch. Try to choose safer *giigoonh*.

LAKE	COUNTY	Women of childbearing age and children less than 15 Maximum number of meals per month	Women beyond childbearing years and men 15 and older Maximum number of meals per month
AMNICON L	DOUGLAS	1	2
ANNABELLE L	VILAS	0	2
BEAR L	ASHLAND	1	2
BIRCH L	VILAS	0	2
BLACK OAK L	VILAS	1	4
BOND L	DOUGLAS	1	4
BUTTERNUT L	PRICE	1	2
CHAIN L	RUSK	1	4
CLAM R FL	BURNETT	Not Enough Information	
CONNORS L	SAWYER	2	4
CRAB L	VILAS	1	2
DAIRYLAND RESERVOIR	RUSK	0	2
DIAMOND L	BAYFIELD	1	2
DOWLING L	DOUGLAS	0	2
DUROY L	PRICE	1	4
ECHO L	IRON	1	4
ENGLISH L	ASHLAND	0	2
FISHER L	IRON	Not Enough Information	
FOREST L	VILAS	1	4
GORDON L	ASHLAND	Not Enough Information	
HARRIS L	VILAS	1	4
HEMLOCK L	BARRON	Not Enough Information	
HIGH L	VILAS	1	4
HOLCOMBE FL	CHIPPEWA	1	4
ISLAND L	RUSK	Not Enough Information	
L MINNESUING	DOUGLAS	0	2
L OF THE FALLS	IRON	Not Enough Information	
L OWEN	BAYFIELD	1	4
L WISSOTA	CHIPPEWA	1	4
LAC SAULT DORE	PRICE	2	4
LONG L	IRON	0	2
LONG L	PRICE	1	4
LONG L	CHIPPEWA	2	8
LOST CANOE L	VILAS	Not Enough Information	
LOST LAND L	SAWYER	1	4
LYNX L	VILAS	0	2
MAMIE L	VILAS	1	4
MIDDLE EAU CLAIRE L	BAYFIELD	1	4
MILLE LACS L	MILLE LACS	2	8
MINONG FL	WASHBURN	1	4
N TURTLE L	VILAS	1	2
NAMEKAGON L	BAYFIELD	1	4
NELSON L	SAWYER	1	4
OXBOW L	VILAS	0	2
PIKE L	PRICE	1	4
PINE L	IRON	1	4
PRESQUE ISLE L CHAIN	VILAS	1	4
RAINBOW FL	ONEIDA	1	2
RIB L	TAYLOR	1	4
ROUND L	PRICE	0	2
S TURTLE L	VILAS	0	2
SISKIWIT L	BAYFIELD	0	2
SOLBERG L	PRICE	0	2
SPIDER L	IRON	Not Enough Information	
SQUAW L	VILAS	1	2
TAINTER L	DUNN	1	4
TEAL L	SAWYER	1	4
TENDERFOOT L	VILAS	1	4
TRUDE L	IRON	0	2
TURNER L	PRICE	1	4
TURTLE-FLAMBEAU FL	IRON	0	2
UPPER EAU CLAIRE L	BAYFIELD	1	4
WHEELER L	OCONTO	1	4
WHITEFISH L	DOUGLAS	1	4

For many native people, *giigoonh* are part of a traditional and healthy diet. If you rely on *giigoonh*, choose safer *giigoonh* with lower levels of mercury by following the advice on this map.

RISKS AND BENEFITS

Risk: Mercury can damage the nervous system, especially the brain. Fetuses and babies are the most at risk because their nervous systems are rapidly developing. Children exposed to unsafe levels while in the womb have been found to experience delayed development in walking and talking, even though the mother was not affected. Mercury cannot be removed by trimming or cooking.

Benefit: Eating even as few as two to three meals of *giigoonh* a month may reduce your risk of death due to heart disease.

If you have questions about finding safer ogaa, call GLIFWC at 1-800-250-7574.
To learn more about mercury in ogaa, visit GLIFWC's website at www.glifwc.org/bio/mercury.htm

Appendix 2

Great Lakes Indian Fish and Wildlife Commission Chain of Custody Forms for Collection and Transport of Fish for Mercury Analysis

FIELD CHAIN-OF-CUSTODY/DATA FORM

Study Title: Spring Walleye Sampling For Mercury **Year:**

Name of Lake:_____ **County**_____ **Area** _____

SECTION A: SAMPLE COLLECTION

COLLECT WALLEYE IN THE FOLLOWING SIZE GROUPS				
Size Ranges	**12.0-14.9**	**15.0-17.9**	**18.0-22**	**>22**
Number of Walleye	**3**	**3**	**3**	**3**

No	Fish Tag No	Length (in.)	Sex (M/F/U)	No	Fish Tag No	Length (in.)	Sex (M/F/U)
1				7			
2				8			
3				9			
4				10			
5				11			
6				12			

SECTION B: SAMPLE STORAGE AND CUSTODY

Check (X) either Cooler or Freezer(<0°C)

1. **Crew Leader/ Warden:**_____ **Date:**_____ **Time:**_____ **Cooler on Ice** _____ **Freezer**_____
2. **Custody given to :** _____ **Date:**_____ **Time:**_____ **Cooler on Ice** _____ **Freezer**_____
3. **Custody given to :** _____ **Date:**_____ **Time:**_____ **Cooler on Ice** _____ **Freezer**_____

Comments:_____

OFFICE USE ONLY– DO NOT WRITE BELOW THIS LINE

---

3. **3rd Custody:** _____ **Date:**_____ **Time:**_____ **Cooler on Ice** _____ **Freezer**_____
4. **4th Custody:** _____ **Date:**_____ **Time:**_____ **Cooler on Ice** _____ **Freezer**_____
5. **5th Custody:** _____ **Date:**_____ **Time:**_____ **Cooler on Ice** _____ **Freezer**_____
6. **6thCustody:** _____ **Date:**_____ **Time:**_____ **Cooler on Ice** _____ **Freezer**_____
7. **7thCustody:** _____ **Date:**_____ **Time:**_____ **Cooler on Ice** _____ **Freezer**_____

TRANSFER CHAIN-OF-CUSTODY FORM

Study Title: Spring Walleye Sampling For Mercury **Year:**

Purpose: Transfer Filets to UW-Superior, LSRI

PAGE 1 of 2

SECTION A: SAMPLE STORAGE

Container Type Enter: 1 = Cooler + Ice 2 = Freezer (≤-10°C)		Placed INTO Container				Taken OUT of Container			
		Date	Time	Initials	°C	Date	Time	Initials	°C
A	GLIFWC	placement into the freezer is recorded on the field COC forms.							
B									
C									
D									
E									
F									

SECTION B: SAMPLE COLLECTION

The individual samples for each lake are listed on the attached sheets.
The lakes being delivered are:

WALLEYE:

1. _____ _____ 11. _____ _____
2. _____ _____ 12. _____ _____
3. _____ _____ 13. _____ _____
4. _____ _____ 14. _____ _____
5. _____ _____ 15. _____ _____
6. _____ _____ 16. _____ _____
7. _____ _____ 17. _____ _____
8. _____ _____ 18. _____ _____
9. _____ _____ 19. _____ _____
10. _____ _____ 20. _____ _____

SECTION C: SAMPLE CUSTODIAN

1. **Collected by:** Collection information list on Field COC at GLIFWC Office.

2. **Transferred by:**_____ **Date:**_____ **Time:**_____

   Relinquished by:_____ **Date:**_____ **Time:**_____

3. **Received by:**_____ **Date:**_____ **Time:**_____

   Relinquished by:_____ **Date:**_____ **Time:**_____

4. **Received by:**_____ **Date:**_____ **Time:**_____

   Relinquished by:_____ **Date:**_____ **Time:**_____

5. **Received by:**_____ **Date:**_____ **Time:**_____

   Relinquished by:_____ **Date:**_____ **Time:**_____

Appendix 3

Comparison of Walleye and Northern Pike Total Mercury Concentrations Analyzed at the University of Wisconsin-La Crosse Mercury Laboratory and the Lake Superior Research Institute

Appendix 3. Comparison of Walleye and Northern Pike Total Mercury Concentrations Analyzed at the University of Wisconsin-La Crosse Mercury Laboratory and the Lake Superior Research Institute.

Lake	Sample ID	UW-LaCrosse ID	Species	Total Length (in)	Sex	LSRI µg Hg/g Dry Wt.	UW-LAX ug Hg/g Dry Wt	RPD	RPA	
Bearskin	9633	ERP-BS04WE012	Walleye	16.7	M	0.584	0.600	2.74	97.3	
Bearskin	9629	ERP-BS04WE006	Walleye	18.6	M	0.842	0.874	3.78	96.2	
Franklin	9620	ERP-FR04WE001	Walleye	25.7	F	1.75	2.24	24.6	75.4	
Franklin	9601	ERP-FR04WE004	Walleye	14.8	M	2.64	0.422	144.8	-44.8	
Mille Lacs	9493	ERP-ML04WE009	Walleye	20.6	F	0.872	0.772	12.2	87.8	
Mille Lacs	9492	ERP-ML04WE007	Walleye	17.3	M	0.777	0.592	27.1	72.9	
Mille Lacs	1374	ERP-ML04NP009	Northern Pike	22.6	M	0.618	0.634	2.49	97.5	
Mille Lacs	1381	ERP-ML04NP012	Northern Pike	39.0	F	0.976	0.889	9.31	90.7	
Squaw	9757	ERP-SQ04WE017	Walleye	16.1	F	2.69	3.03	12.0	88.0	
Squaw	9653	ERP-SQ04WE008	Walleye	14.2	M	2.36	3.17	29.3	70.7	
Tomahawk Chain	9684	ERP-TH04WE002	Walleye	19.9	F	1.09	1.07*	2.02	98.0	
Tomahawk Chain	9693	ERP-TH04WE007	Walleye	24.0	M	2.86	3.06	6.67	93.3	
								23.1	**76.9**	**Mean**
								39.6	**39.6**	**St Dev**

RPD = Relative Percent Difference
RPA = Relative Percent Agreement
* Value is average of triplicate measurements

8 out of 12 samples were above 80% relative agreement
11 out of 12 samples were above 70% relative agreement

Appendix 4

Quality Assurance Report: 2004 Field Data Collection for EPA Grant # 96540801-0

Quality Assurance Report: 2004 Field data collection for EPA Grant # 96540801-0

By:

Matt Hudson
Environmental Biologist
Great Lakes Indian Fish and Wildlife Commission
Field Manager, EPA Grant # 96540801-0

Introduction

The following report satisfies quality assurance reporting requirements outlined in section 14.1 of the Quality Assurance Project Plan entitled "Great Lakes Indian Fish and Wildlife Commission (GLIFWC) Testing of Fish for Mercury Using EPA Supplemental Funds - EPA Grant # 96540801-0".

Quality Assurance Summary

1. System and Performance Audits - Results from the field audit, which included an audit of field walleye collections and an audit of GLIFWC laboratory tissue processing and data collection, are described in Appendix A. In general, protocols for data collection and sample handling were followed well by staff observed during the audits. Minor comments were made on improving the completion of chain of custody forms, but no major problems or deviations were noted.

2. Completeness and Quality of Field Sampling Process and Data - Funds were available to analyze 300 walleye for mercury from 25 lakes in 2004 under EPA Grant # 96540801-0. Plans called for twelve walleye to be collected, with three fish taken from each of four size ranges (12.0 to 14.9, 15.0 to 17.9, 18.0 to 22.0, and greater than 22.0 inches). Because twelve fish are not typically collected from all lakes, additional lakes were selected to reach the goal of 300 fish. A total of 35 lakes were selected for sampling and a total of 331 walleye samples from 33 lakes were collected (Table 1). The Lake Superior Research Institute (LSRI) reported mercury results from 332 samples. A walleye labeled with "10833/10832" was analyzed by LSRI, but no field record for the collection of this fish was available. Therefore, the reported concentration for this fish was not entered into GLIFWC's mercury database.

Overall, sample collection and analysis exceeded project goals. Observed collection of field samples and tissue processing and data collection was adequately followed according to QAPP guidelines. Therefore, no problems are seen with the quality of field data for this project.

3. Deviations - One deviation form was completed (Appendix B). The deviation did not affect the quality of the data or the data collection process, so no corrective action was necessary.

4. Significant Quality Assurance Problems and Recommended Solutions - No significant quality assurance problems were noted during the 2004 field sample and data collection process.

Table 1. Summary of completeness of mercury walleye collections during spring 2004 as part of EPA Grant # 96540801-0.

Lake Name	County	State	Size Group				Collection Goal	Total Collected	Percent of Goal
			12.0 to 14.9	15.0 to 17.9	18.0 to 22.0	> 22.0			
Middle Eau Claire L	Bayfield	WI	3	3	3	3	12	12	100%
Upper Eau Claire L	Bayfield	WI	3	3	3	3	12	12	100%
L Nebagamon	Douglas	WI	3	3	3	3	12	12	100%
Upper St. Croix L	Douglas	WI	3	3	3	1	12	10	83%
Roberts	Forest	WI	3	3	3	1	12	10	83%
L Lucerne	Forest	WI	3	3	5	1	12	12	100%
L Gogebic	Gogebic	MI	3	0	6	3	12	12	100%
Tutle-Flambeau Fl	Iron	WI	3	3	1	0	12	7	58%
L Nokomis (Rice R Fl)	Lincoln	WI	3	3	3	1	12	10	83%
Sevenmile L	Oneida	WI	3	4	4	2	12	13	108%
Pelican L	Oneida	WI	3	3	4	2	12	12	100%
Balsam L	Polk	WI	0	2	1	0	12	3	25%
L Chippewa	Sawyer	WI	3	3	3	3	12	12	100%
Round L	Sawyer	WI	3	3	3	2	12	11	92%
Nelson L	Sawyer	WI	1	3	3	1	12	8	67%
Spider L	Sawyer	WI	3	3	3	3	12	12	100%
L Laura	Vilas	WI	3	3	3	3	12	12	100%
High L	Vilas	WI	3	3	3	3	12	12	100%
Island L	Vilas	WI	3	4	2	1	12	10	83%
Manitowish L	Vilas	WI	5	4	3	0	12	12	100%
Alder L	Vilas	WI	3	3	1	1	12	8	67%
Crab L	Vilas	WI	3	3	2	1	12	9	75%
Kentuck L	Vilas	WI	0	0	0	0	12	0	0%
Rest L	Vilas	WI	3	3	2	0	12	8	67%
Big Portage	Vilas	WI	3	3	1	0	12	7	58%
Little Star L	Vilas	WI	3	4	5	0	12	12	100%
Clear L	Vilas	WI	2	3	1	0	12	6	50%
Sherman L	Vilas	WI	3	3	3	3	12	12	100%
Spider L	Vilas	WI	7	3	0	0	12	10	83%
Long L	Vilas	WI	4	2	6	0	12	12	100%
Ballard L	Vilas	WI	0	1	0	0	12	1	8%
Lac Vieux Desert	Vilas	WI	3	3	0	0	12	11	92%
Wild Rice L	Vilas	WI	8	3	0	0	12	11	92%
Bass-Patterson L	Washburn	WI	3	3	3	1	12	10	83%
		Total Collected	102	96	86	42	408	331	81%

Appendix A

Field audit of walleye collections and tissue processing data collection for EPA Grant # 96540801-0

Field Audit Form

Section 1: Data Collection

Data Type	(+/-)[a]	Comments	Date Observed
Sample Storage & Transfer	+	Fish kept in cooler in field, transferred to freezer @ hotel	4/29/04
Length/Sex/Tag	+	Excellent	↓
Fill out COC	+	All required data included and legible	↓
Age[b]			

[a]: + = in compliance, - = out of compliance

[b]: Age will be determined at lab and not in the field.

General Comments:

I was present on Butch M.'s assessment boat the night of 4/29/04 on Franklin Lake, Forest Co. The crew effectively and efficiently collected samples for mercury while following instructions for the collection. No problems seen.

Section 2: Tissue Collection

Data Type	(+/-)[a]	Comments	Date Observed
Fillet Removal	+	Excellent	4/13/04
Fill out datasheet	+	↓	↓

[a]: + = in compliance, - = out of compliance

General Comments:

Procedures were followed and no concerns related to data collection or contamination of samples were seen. Tissue collection was done in the lab at GLIFWC by either EK or SC throughout the collection season.

Section 3: Sample Packaging

Data Type	(+/-)[a]	Comments	Date Observed
Lab processing	+	Label included - processed samples kept separate in freezer from unprocessed	4/13/04
field collection	+	According to protocal	4/29/04

[a]: + = in compliance, - = out of compliance

General Comments:

Section 4: Storage

Data Type	(+/-)[a]	Temp (°C)[b]	Comments	Date Observed
freezer temp. record	+	-18	No glaring problems	4/24/04

[a]: + = in compliance, - = out of compliance
[b]: Temperature of storage container

General Comments: Freezer at the hotel in Eagle River allowed the crews to put walleye in freezer immediately. temperature records on the freezer were kept satisfactorily

Section 5: Custody (Chain-of-Custody Forms)

Data Type	(+/-)[a]	Comments	Date Observed
Tag #, length, Sex	+	Only comment was to remember to fill out the date and type of container for transfer	4/29/04

[a]: + = in compliance, - = out of compliance

General Comments:

Section 6: Transport

Data Type	(+/-)[a]	Comments	Date Observed
Field collection + transport	+	Cooler on ice - according to protocol	4/29/04

[a]: + = in compliance, - = out of compliance

General Comments:

Auditor Name: Matt Hudson

Auditor Signature: Matt Hudson Date Signed: 6/4/04

Appendix B

Deviation forms for EPA Grant # 96540801-0

DEVIATION FORM - GREAT LAKES INDIAN FISH AND WILDLIFE COMMISSION

Project Title: Testing of fish for mercury using EPA supplemental funding **Date/Time:** 7/6/2005

Explanation of Deviation: Section 12.2, pg 34 of QAPP - Statistics including analysis of variance (ANOVA), checking assumptions of an ANOVA, and a Dunnett's test will not be calculated as stated in the QAPP.

Corrective Procedure: None required. Calculation of these statistics is not necessary as part of the data quality assessment for this project.

Signature: Matt Ho Date: 7/6/05

Route to Project Manager for Evaluation.

Impact on this Study:

NONE

X Signature: Date: 7/27/05

Appendix 5

Lake Superior Research Institute Laboratory Quality Assurance Audit Report on the Spring 2004 Walleye Project

Quality Assurance Audit Report on the Spring 2004 Walleye Project

Audit Date: July 2005
Report Date: July 20, 2005

Auditor: Dianne Brooke

(A) Description and Scope of Audit

As part of a contaminant environmental monitoring study that was begun due to increased concerns about health risks and the consumption of fish, LSRI biologists and chemists are analyzing fish samples for contaminant levels. This audit report contains a review of the sample processing methodology, data recording, data entry, and QA/QC training exercises. The sample processing methodology for the Spring 2004 Walleye Project (date of contract = May 19 - December 2004) was not observed by the LSRI QA Manager. However, a similar project conducted in the spring of 2004 where lake trout samples were processed and composited, contained an internal QA/QC audit completed by the LSRI QA/QC Manager. This audit outlines the QA/QC observations for both the Spring 2004 Lake Trout and Walleye Projects. The findings for each project are listed under the subheadings.

(B) Major Findings

Spring 2004 Lake Trout Project (Processing Methodology)

On April 28, 2004, Dianne Brooke (LSRI QA Manager) observed three staff and three student personnel processing the lake trout samples. Fish Number 1431 was being filleted when the audit began. The following observations were made and discussed with the project staff.

- All personnel wore lab coats and gloves, but some individuals were not wearing safety glasses.
- Only one of the three students had a record of formal project SOP training, while the other two students were informally trained (i.e., they did not read through the entire SOP, but project staff had demonstrated portions of the methodologies to them).
- The three staff members and three students had received training in Good Laboratory Practices. Training certificates were on file for the six project personnel.
- The data bench sheets (i.e., Lake Trout Processing Form, Lake Trout Composite Preparation Form and Percent Moisture Content of Composite Form) for the 4/28/04 processing date did not contain the study ID number (each separate study has a unique number assigned to it).
- Some of the columns for the Lake Trout Composite Preparation Form (skin) and Percent Moisture Content of Composite Tissues (wet tissue weight, dry tissue

weight, and percent moisture) were not filled out (if there were no data entries, a line should be drawn through the column).

- A column should be added on the processing bench sheets for the initials of the person weighing the various components of the fish body.
- On the Lake Trout Processing Form, information for the daily balance check could be added to the bench sheets, thus the data recorder would have to enter only the balance readings.
- The sample labeling for the composites was well done; they were clearly written and utilized different colored tape.
- I observed staff members taring the balance in between the weighing of samples, and the bowls containing the composite were mixed for a minimum of three minutes.

In discussion with project staff and based on some of the observations of the sample processing methodology, two SOPs were developed (*SA/45 - Processing Several Fish into Three Homogeneous Composites of Different Tissue Type* and *SA/46 - Processing Several Fish into One Homogeneous Fish Composite*).

Spring 2004 Walleye Project - Notebooks/Bench Sheets

Reviewed the three-ring binder entitled *GLIFWC Spring Walleye 2004* and *Vol. 02-09-25-HS GLIFWC.*

- The binder was somewhat confusing in its progression of contents. More detailed ringbook indexes, listing dates and sample lakes would be helpful. The QAPP, chain-of-custody records, instrument data output, and QA/QC data would present a logical order of the information.
- On the QA data forms, a unit designation is needed for the "g sample" and column headings for the detection limit assumptions.
- The study ID number should appear on all output sheets.
- The data in the binder appeared to be thoroughly proofed, both for entry errors and calculation errors. The person checking the data initialed the rechecks and recorded the date when the data was proofed.
- In analyzing the samples for tissue moisture analyses, approximately 58% (203/351 samples) were chosen for this parameter. Of the 203 samples, 10.8% were analyzed in duplicate and checked for percent agreement. The percent duplicate agreement for tissue moisture analyses ranged from 99.3 - 99.8%. Of the 203 samples, 10% were placed back into the oven and reweighed after an additional 24 hours to ensure dryness. The QA/QC drying exercise yielded values that were above 99.0% duplicate agreement.
- Typically an analysis set consists of 40 samples being analyzed for mercury content. For each data set, the following QA/QC samples were analyzed: two dorm samples in duplicate, four duplicate agreement samples, and four spike

recovery samples in duplicate. A calibration blank and five standards were also analyzed with the data set. One set of standards was run at the beginning of the analyses and the other set interspersed throughout the sampling series. This was recorded on preprinted bench sheets for the analyses dates of: 6/22/04, 6/24/04 (which had to be rerun due to high percent recoveries for the dorm samples), and 7/01/04. In briefly looking at the other dates of analyses, the same formats of QA/QC samples were used.

- For the 10/13/04 sample analysis date, seven additional samples were analyzed to check for percent duplicate agreement. The reason for the re-analyses was the first set of percent duplicate agreement samples did not pass the QA/QC criteria. Those same samples were re-analyzed in duplicate and passed QA/QC criteria the second time.
- The lab notebook *Vol. 02-09-25-HS GLIFWC* was well organized and contained clearly recorded data entries. It was missing the Table of Contents entries and a tabular index to delineate the separate studies that were documented in the notebook.
- The method SOPs were referred to in the lab notebook and recording errors were properly crossed-out, dated, and initialed.
- An entry on page 48 dated 6/28/04 stated that the sample set failed QA, but it did not contain an explanation.
- A couple entries did not have the initials of the person recording the data. Periodically, the principal investigator should review the recorded analyses' data and sign/date the entry of review.

3. Recommendations

The overall reviews of the methodology and data recording indicate that study personnel are highly organized and intentional in their QA/QC protocols for conducting research. The SOPs for the project are continually being revised and new ones are being written when a need arises. One concern, based on the audit, is that there are a number of small/similar environmental monitoring studies that are being conducted back-to-back. There should be clear delineations of when one study stops and the other starts. This may be best accomplished by assigning a unique study ID number that is used on all output for the project. A brief description of the study should be written in the lab notebook at the onset of analyses (it would include the number of fish, sample lakes, personnel involved, contract number, project dates, sample collection methodology, and the list of SOPs needed to complete the project). More complete documentation for SOP training needs should be coordinated between the principal investigator and the LSRI QA Manager.

Appendix 6

2002 Total Mercury Data in Muskellunge Analyzed by the Wisconsin State Laboratory of Hygiene

Appendix 6. 2002 Total Mercury in Muskellunge Data Analyzed by the Wisconsin State Laboratory of Hygiene.

Site Name	County Name	Collection Date	Sample Collector Name	Sample ID	Species	Fish Form Name	Sex	Fish Length (in)	Total Mercury (ug/g)	LOD	LOQ
Big Lake	Vilas	4/30/2002	GLIFWC	TAG 11583	MUSKELLUNGE	EDIBLE PORTION	U	33.8	0.9	0.004	0.013
Big Lake	Vilas	4/30/2002	GLIFWC	TAG 11581	MUSKELLUNGE	EDIBLE PORTION	U	37.0	1.1	0.004	0.013
Big McKenzie Lake	Burnett	5/10/2002	GLIFWC	TAG 11512	MUSKELLUNGE	EDIBLE PORTION	M	31.3	0.31	0.004	0.013
Big McKenzie Lake	Burnett	5/10/2002	GLIFWC	TAG 11513	MUSKELLUNGE	EDIBLE PORTION	M	35.6	0.55	0.004	0.013
Big McKenzie Lake	Burnett	5/10/2002	GLIFWC	TAG 11511	MUSKELLUNGE	EDIBLE PORTION	U	37.0	0.78	0.004	0.013
Big McKenzie Lake	Burnett	5/10/2002	GLIFWC	TAG 11514	MUSKELLUNGE	EDIBLE PORTION	U	38.0	0.64	0.004	0.013
Big McKenzie Lake	Burnett	5/10/2002	GLIFWC	TAG 11515	MUSKELLUNGE	EDIBLE PORTION	F	40.0	0.75	0.004	0.013
Big St Germain Lake	Vilas	4/30/2002	GLIFWC	TAG 11588	MUSKELLUNGE	EDIBLE PORTION	F	41.5	0.34	0.004	0.013
Little St Germain Lake East Bay	Vilas	4/30/2002	GLIFWC	TAG 11598	MUSKELLUNGE	EDIBLE PORTION	M	31.0	0.086	0.004	0.013
Little St Germain Lake East Bay	Vilas	4/30/2002	GLIFWC	TAG 11597	MUSKELLUNGE	EDIBLE PORTION	M	34.6	0.17	0.004	0.013
Little St Germain Lake East Bay	Vilas	4/30/2002	GLIFWC	TAG 11594	MUSKELLUNGE	EDIBLE PORTION	U	37.0	0.19	0.004	0.013
North Twin Lake Chain	Vilas	4/28/2002	GLIFWC	TAG 11602	MUSKELLUNGE	EDIBLE PORTION	F	34.7	0.52	0.004	0.013
North Twin Lake Chain	Vilas	4/28/2002	GLIFWC	TAG 11601	MUSKELLUNGE	EDIBLE PORTION	F	44.7	0.97	0.004	0.013
Squirrel Lake	Oneida	4/30/2002	GLIFWC	TAG 11539	MUSKELLUNGE	EDIBLE PORTION	F	39.0	0.62	0.004	0.013
Yellow Lake	Burnett	4/22/2002	GLIFWC	TAG 11525	MUSKELLUNGE	EDIBLE PORTION	U	31.1	0.25	0.004	0.013

Appendix 7

Lake Superior Research Institute Final Report: Total Mercury in Walleye and Muskellunge Muscle Tissue Captured in the Ceded Territories During the Spring of 2003

Total Mercury in Walleye and Muskellunge Muscle Tissue Captured in the Ceded Territories During the Spring of 2003

for

Great Lakes Indian Fish and Wildlife Commission
P.O. Box 9
Odanah, Wisconsin 54861

by

Larry T. Brooke
Christine N. Polkinghorne
Heidi J. Saillard
Thomas P. Markee

Lake Superior Research Institute
University of Wisconsin-Superior
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October 2003

Introduction

Filets of muscle tissue from walleye (*Stizostedion vitreum*) and muskellunge (*Esox masquinongy*) captured during the spring of 2003 were delivered to the Lake Superior Research Institute (LSRI) of the University of Wisconsin-Superior for analysis of mercury content. Walleye were collected from sixteen lakes while muskellunge were collected from eight lakes. All lakes sampled are within the 1837 and 1842 Treaty ceded territories. The analyses were conducted during May through August 2003.

Methods

At the time fish were captured, a tribal warden or biologist was present to measure the total length of each fish. The fish were tagged with a unique number (i.e., a fish identification number) and whole fish with chain-of-custody forms were transferred to the Great Lake Indian Fish and Wildlife Commission (GLIFWC) laboratory. The samples were immediately placed on ice and were frozen within 36 hours of capture. At the GLIFWC laboratory, one or two filets were removed from each fish, the skin removed and the fish identification number and skinless filet was placed into a plastic bag (Appendix A). Sex of the fish was determined during the fileting process. A dorsal fin spine was removed from each fish to determine its age. At the LSRI laboratories, the walleye were received frozen and in good condition with chain-of-custody documentation. The samples were stored in a freezer at approximately -18 C until they were removed and thawed for processing and analysis.

Before processing of the fish tissues, all glassware, utensils, and grinders were cleaned according to the appropriate methods (Appendices B and C). Each day, the fish that would be processed were removed from the freezer and allowed to warm to a flexible, but stiff, consistency. The skinless filet was ground three times in a grinder. A small amount of the initial tissue that passed thorough the grinder was collected and discarded (Appendix D). A sub-sample of the ground tissue was placed into a critically cleaned glass vial and frozen until the mercury analysis was conducted. The grinder was disassembled after each filet was ground and the unit was washed according to the grinder cleaning procedure (Appendix C).

Fish tissues were weighed for mercury analysis following standard laboratory procedure (Appendix E). Mercury solutions for making tissue spikes and preparing analytical standards were prepared by the procedures in Appendix F. Mercury analyses were performed using cold vapor mercury analysis techniques on a Perkin Elmer FIMS 100 mercury analysis system (Appendix G). The method has a biota detection limit of 0.00188 μg Hg/g for a tissue mass of 0.1 g.

Moisture content of tissue was measured by difference of the wet tissue weight and the dried tissue weight (Appendix H). A portion (1 to 4 g) of ground tissue was placed into a dry and weighed aluminum drying pan immediately following tissue grinding. The pan and wet tissue were immediately weighed and placed into an oven (60 C) and dried for various time intervals.

Drying times varied from 24 to 96 hours. Approximately 47% of the walleye analyzed for mercury had moisture content determined. In addition, moisture contents were measured in all of the muskellunge filets.

Quality Assurance

Quality of analysis was monitored by four methods: Analysis of similar fish tissues before and after the tissue grinding process (procedural blanks) to measure laboratory bias; analysis of dogfish shark (DORM-2, *Squalus acanthias*) from the Canadian government (certified reference material from National Research Council Canada, Ottawa, Ontario, Canada) that has a certified concentration of mercury to measure analytical accuracy; duplicate analysis of fish tissue from the same filet to measure analytical precision; and analysis of tissue with known additions of mercury to determine analytical interferences. Two sets of standard solutions with known amounts (analytical standards) of mercury were analyzed with each group (maximum of 40 samples plus QA samples) of tissue samples. These analytical solutions contained 0, 50, 100, 500, 1000 and 6000 ng Hg/L. They were prepared from a purchased 1000 ± 10 ppm mercury (made with mercuric nitrate) reference standard solution (Environmental Research Associates, Arvada, CO).

Duplicate agreement and spike recovery values were acceptable when in the range of >80.9% for duplicate agreement and 57.1 to 112.7% for spike recovery. All acceptable ranges are calculated as mean ± 2 times the standard deviation of all analyses of the appropriate samples conducted from 6/12/02 to 4/4/03 at the LSRI laboratory.

A commercial canned tuna fish (*Thunnus sp.*) sample was used as a measurement of laboratory bias on the grinding process for sample preparation. One portion of each can was transferred directly into a sample bottle after the liquid was squeezed out of the can. The second portion was ground in the same manner as the walleye filets. This check was made to ensure that no contamination or loss of mercury was occurring in the grinding process. Analysis of the canned tuna fish from three occasions coincident with the grinding of walleye resulted in a mean of 87.1 ± 12.2 percent agreement (Table 1) for mercury analysis.

Analysis of the dogfish shark tissue (DORM-2) of certified concentration was conducted with each set of walleye and muskellunge tissues analyzed (Table 2). The certified mercury concentration for the dogfish tissue was 4.64 ± 0.26 µg Hg/g. The grand mean and standard deviation for the analyses of the dogfish shark tissue during this study at the LSRI laboratory was 4.39 ± 0.32 µg Hg/g (5.4% less than the certified value). An acceptable range of mercury concentrations was calculated for this study based upon the analyses conducted from 6/12/02 to 4/4/03 (mean ± 2 times the standard deviation of all analyses). The calculated acceptable range was 3.61 to 4.84 µg Hg/g. One of the DORM-2 tissue sets analyzed on 7/15/03 was outside of the accepted limit. However, the average of the DORM-2 analyses for the day was 96.8%, therefore making the data for that set acceptable.

Fish tissues were analyzed in duplicate twenty-two times. Two portions of the same tissue were analyzed independently. Agreement between two mercury analyses of the same tissue averaged 93.1 ± 4.79 percent (Table 3). Duplicate agreement is considered acceptable when it is >80.9 percent based upon the mean ± 2 times the standard deviation of all analyses of the duplicate samples conducted from 6/12/02 to 4/4/03.

Prior to digestion, tissues from 22 fish samples were spiked, in duplicate, with a known quantity of mercury and analyzed for recovery of the spiked mercury (Table 4). Spike recovery is considered acceptable when it is in the range of 57.1 to 112.7% of the expected value. This is based upon the mean ± 2 times the standard deviation of all analyses of the spiked samples conducted from 6/12/02 to 4/4/03. Grand mean and standard deviation of the spike recovery for all samples was 84.4 ± 13.6 percent.

Results

Mercury Analysis – Skinless filets of 165 walleye from 15 lakes and 17 muskellunge from 8 lakes in Wisconsin were analyzed for total mercury content. Total mercury concentrations on a wet weight basis ranged from 0.079 to 1.61 µg Hg/g (parts per million) in muscle tissue from the samples (Table 5). In addition, skinless filets of 4 walleye from 1 Michigan lake were analyzed for total mercury content. Total mercury concentrations on a wet weight basis ranged from 0.955 to 1.42 µg Hg/g (parts per million) in muscle tissue from the samples (Table 5). Concentrations tended to vary within a lake by size (larger fish having higher mercury concentrations) and between lakes for similar size groups.

Tissue Moisture Analysis – Moisture was measured in the muscle of ground filets immediately following grinding (Table 7). Walleye muscle tissue contained 79.4 ± 1.02 percent water determined in a sample of 47 percent of the 169 analyzed filets. Seventeen muskellunge from eight lakes had moisture determinations with a mean value of 76.2 ± 1.64 percent.

Table 1. Percent Agreement of Procedural Blank Samples [Commercial Tuna Fish (*Thunnus* sp.) Before and After Grinding] for Total Mercury.

Date of Analysis	Grinding Date	Before Grinding (µg Hg/g)	After Grinding (µg Hg/g)	Percent Agreement
7/3/03	6/13/03	0.025	0.028	89.3
7/9/03	6/24/03	0.139	0.188	73.9
7/29/03	7/23/03	0.052	0.051	98.1

Table 2. Mercury Concentrations of Dogfish Shark (*Squalus acanthus*) Tissue Supplied by the National Research Council Canada (DORM-2). The Tissue has a Certified Mercury Concentration of 4.64 ± 0.26 µg Hg/g Tissue.

Date Analyzed	Sample 1	Sample 2	Mean	Std. Dev.	Percent of Expected
7/3/03	4.32	4.35	4.33	0.02	93.4
7/3/03	4.60	-[a]	4.60	-	99.1
7/9/03	4.30	4.56	4.43	0.18	95.4
7/9/03	4.00	5.29	4.65	0.91	100.2
7/15/03	3.88	3.80	3.84	0.06	82.8
7/15/03	5.13	5.14	5.14	0.01	110.7
7/23/03	4.25	4.22	4.24	0.02	91.3
7/23/03	4.44	-[a]	4.44	-	95.7
7/29/03	4.54	4.38	4.46	0.11	96.1
7/29/03	4.38	4.31	4.34	0.05	93.6
8/12/03	4.05	3.99	4.02	0.04	86.6
8/12/03	4.24	4.36	4.30	0.08	92.7

[a] Instrument malfunction during analysis.

Table 3. Percent Agreement Between Duplicate Analysis for Total Mercury (Wet Weight) Content in Skinless Filet Tissue of Walleye and Muskellunge Captured from Ceded Territories Inland Waters during 2003.

Date of Analysis	Lake	Analysis 1 (µg Hg/g)	Analysis 2 (µg Hg/g)	Percent Agreement
	Walleye			
7/3/03	Bass Patterson 6685	0.396	0.425	93.2
7/3/03	North Twin 6777	0.285	0.262	91.9
7/3/03	Sherman 6502	0.303	0.330	91.8

7/3/03	Sherman 6696	0.268	0.286	93.7
7/9/03	Owen 6534	0.647	0.731	88.5
7/9/03	Owen 6538	0.458	0.504	90.8
7/9/03	Anabelle 6626	0.737	0.767	96.2
7/9/03	Siskiwit 6638	0.699	0.820	85.3
7/15/03	Willow 6755	0.515	0.517	99.5
7/15/03	Squirrel 6602	0.194	0.192	99.4
7/15/03	Gile Flowage 6512	0.772	0.863	89.5
7/15/03	Butternut 6670	0.150	0.154	97.2
7/23/03	Chippewa Flowage 6724	0.191	0.181	94.9
7/23/03	Chippewa Flowage 6730	0.214	0.203	95.0
7/23/03	Kentuck 6659	0.202	0.219	92.3
7/29/03	Namekagon 6738	0.524	0.536	97.7
7/29/03	Namekagon 6745	0.364	0.328	90.2
7/29/03	Turtle Flambeau Flowage 6710	0.548	0.630	87.0
7/29/03	Minocqua 6771	0.256	0.247	96.5
	Muskellunge			
8/12/03	Yellow 0343	0.654	0.802	81.6
8/12/03	Minocqua 0306	0.438	0.437	99.8
8/12/03	North Twin 0338	0.536	0.558	96.1

Table 4. Percent of Mercury Recovered from Skinless Walleye and Muskellunge Filet Samples Spiked with a Known Quantity of Mercury Coincident with the Analysis of Walleye and Muskellunge (2003).

Date of Analysis	Lake	Spike #1	Spike #2	Mean	Std Dev.
	Walleye				
7/3/03	Bass Patterson 6685	68.5	83.9	76.2	10.9
7/3/03	North Twin 6777	82.0	90.8	86.4	6.22

7/3/03	Sherman 6502	92.1	84.5	88.3	5.41
7/3/03	Sherman 6696	91.0	85.5	88.2	3.94
7/9/03	Owen 6534	86.4	79.6	83.0	4.84
7/9/03	Owen 6538	80.5	83.7	82.1	2.27
7/9/03	Anabelle 6626	81.8	71.7	76.7	7.12
7/9/03	Siskiwit 6638	47.3	68.4	57.9	14.9
7/15/03	Willow 6755	80.6	81.7	81.2	0.78
7/15/03	Squirrel 6602	97.3	90.5	93.9	4.79
7/15/03	Gile Flowage 6512	69.2	60.8	65.0	5.95
7/15/03	Butternut 6670	96.2	95.1	95.6	0.81
7/23/03	Chippewa Flowage 6724	94.7	83.2	89.0	8.13
7/23/03	Chippewa Flowage 6730	113.0	90.4	101.7	15.9
7/23/03	Kentuck 6659	86.5	91.9	89.2	3.86
7/29/03	Namekagon 6738	100.5	76.6	88.5	16.9
7/29/03	Namekagon 6745	90.1	99.2	94.6	6.48
7/29/03	Turtle Flambeau Flowage 6710	115.0	84.8	99.9	21.4
7/29/03	Minocqua 6771	112.4	113.4	112.9	0.74
	Muskellunge				
8/12/03	Yellow 0343	51.9	66.1	59.0	10.0
8/12/03	Minocqua 0306	79.2	78.4	78.8	0.58
8/12/03	North Twin 0338	74.3	63.1	68.7	7.96

Table 5. Total Mercury Concentrations (Wet Weight) in Walleye and Muskellunge Filets from Various Length and Sex Fish Captured during the Spring of 2003.

Lake	Sample ID	Date Analyzed	Field Length (in)	Sex	µg Hg/g
		Walleye			
Annabelle	6621	7/9/2003	27.3	F	1.610

Annabelle	6622	7/9/2003	12.8	M	0.541
Annabelle	6623	7/9/2003	12.5	M	0.686
Annabelle	6624	7/9/2003	24.0	F	0.843
Annabelle	6625	7/9/2003	13.2	F	0.572
Annabelle	6626	7/9/2003	18.3	F	0.752
Annabelle	6627	7/9/2003	16.7	M	0.989
Annabelle	6628	7/9/2003	13.1	M	0.754
Annabelle	6629	7/9/2003	15.8	F	0.850
Annabelle	6630	7/9/2003	13.3	F	0.596
Annabelle	6632	7/9/2003	16.8	F	0.764
Annabelle	6635	7/9/2003	11.9	M	0.520
Bass-Patterson	6681	7/3/2003	27.0	F	0.701
Bass-Patterson	6682	7/3/2003	12.7	M	0.209
Bass-Patterson	6683	7/3/2003	12.7	M	0.181
Bass-Patterson	6684	7/3/2003	14.2	M	0.273
Bass-Patterson	6685	7/3/2003	15.1	M	0.411
Bass-Patterson	6686	7/3/2003	15.1	M	0.374
Bass-Patterson	6687	7/3/2003	17.6	M	0.376
Bass-Patterson	6688	7/3/2003	24.0	F	0.740
Bass-Patterson	6689	7/3/2003	20.4	F	0.471
Bass-Patterson	6690	7/3/2003	16.8	M	0.269
Bass-Patterson	6691	7/3/2003	19.2	M	0.296
Bass-Patterson	6692	7/3/2003	22.4	F	0.560
Butternut	6666	7/15/2003	13.1	M	0.0863
Butternut	6667	7/15/2003	23.7	F	0.474
Butternut	6668	7/15/2003	20.5	F	0.249
Butternut	6669	7/29/2003	20.2	F	0.228
Butternut	6670	7/15/2003	17.4	M	0.152

Butternut	6671	7/15/2003	13.6	M	0.0785
Butternut	6672	7/15/2003	22.4	F	0.251
Butternut	6673	7/15/2003	17.8	M	0.220
Butternut	6676	7/15/2003	21.2	F	0.305
Butternut	6677	7/15/2003	22.7	F	0.278
Butternut	6679	7/15/2003	15.5	M	0.131
Butternut	6680	7/15/2003	13.1	M	0.0813
Chippewa Fl	6717	7/23/2003	19.2	F	0.584
Chippewa Fl	6718	7/23/2003	21.2	F	0.324
Chippewa Fl	6720	7/23/2003	16.0	M	0.408
Chippewa Fl	6721	7/23/2003	23.3	F	0.409
Chippewa Fl	6722	7/23/2003	14.7	M	0.546
Chippewa Fl	6723	7/23/2003	15.5	M	0.290
Chippewa Fl	6724	7/23/2003	15.8	M	0.186
Chippewa Fl	6725	7/23/2003	24.9	F	0.780
Chippewa Fl	6726	7/23/2003	19.0	F	0.292
Chippewa Fl	6727	7/23/2003	22.6	F	0.972
Chippewa Fl	6729	7/23/2003	12.9	M	0.191
Chippewa Fl	6730	7/23/2003	13.7	M	0.209
Gile Fl	6511	7/15/2003	16.5	M	0.719
Gile Fl	6512	7/15/2003	18.6	F	0.818
Gile Fl	6514	7/15/2003	15.1	M	0.570
Gile Fl	6518	7/15/2003	13.6	M	0.410
Gile Fl	6520	7/23/2003	18.0	F	0.871
Gile Fl	6523	7/15/2003	14.5	M	0.464
Gile Fl	6524	7/15/2003	15.5	M	0.716
Gile Fl	6525	7/15/2003	17.0	F	0.686
Kentuck	6652	7/23/2003	15.3	M	0.204

Kentuck	6653	7/23/2003	14.4	M	0.211
Kentuck	6655	7/23/2003	14.0	M	0.173
Kentuck	6656	7/23/2003	13.0	M	0.148
Kentuck	6659	7/23/2003	15.7	M	0.211
Kentuck	6660	7/23/2003	15.4	M	0.141
Minocqua	6761	7/23/2003	20.3	F	0.353
Minocqua	6763	7/23/2003	15.8	M	0.264
Minocqua	6764	7/29/2003	19.5	M	0.673
Minocqua	6765	7/29/2003	26.6	F	0.822
Minocqua	6766	7/29/2003	24.5	F	0.658
Minocqua	6768	7/29/2003	16.0	M	0.358
Minocqua	6770	7/29/2003	18.5	M	0.245
Minocqua	6771	7/29/2003	14.5	M	0.251
Minocqua	6772	7/29/2003	23.5	F	0.362
Minocqua	6773	7/29/2003	14.7	M	0.154
Minocqua	6774	7/29/2003	16.2	M	0.420
Minocqua	6775	7/29/2003	14.5	M	0.189
Namekagon	6731	7/29/2003	26.0	F	0.670
Namekagon	6732	7/29/2003	19.3	F	0.305
Namekagon	6733	7/29/2003	16.2	M	0.304
Namekagon	6735	7/29/2003	14.1	M	0.193
Namekagon	6736	7/29/2003	17.6	F	0.305
Namekagon	6737	7/29/2003	18.8	M	0.528
Namekagon	6738	7/29/2003	22.6	F	0.530
Namekagon	6739	7/29/2003	14.5	M	0.312
Namekagon	6741	7/29/2003	15.0	M	0.300
Namekagon	6743	7/29/2003	18.1	F	0.547
Namekagon	6744	7/29/2003	12.5	M	0.568

Namekagon	6745	7/29/2003	17.8	M	0.364
Owen	6526	7/9/2003	25.7	F	0.769
Owen	6527	7/9/2003	13.0	M	0.184
Owen	6528	7/9/2003	20.8	F	0.732
Owen	6529	7/9/2003	22.7	F	0.387
Owen	6530	7/9/2003	16.2	M	0.185
Owen	6531	7/9/2003	14.3	M	0.198
Owen	6534	7/9/2003	24.2	F	0.689
Owen	6535	7/9/2003	16.9	M	0.264
Owen	6536	7/9/2003	14.5	M	0.184
Owen	6537	7/9/2003	19.6	M	0.391
Owen	6538	7/9/2003	18.6	F	0.481
Owen	6539	7/9/2003	16.4	M	0.295
Pike Lake (MI)	6545	8/11/2003	23.4	M	0.955
Pike Lake (MI)	6550	8/11/2003	21.4	M	1.42
Pike Lake (MI)	6664	8/11/2003	21.1	U	1.36
Pike Lake (MI)	6678	8/11/2003	19.7	M	1.12
Sherman	6501	7/3/2003	14.8	M	0.264
Sherman	6502	7/3/2003	18.2	M	0.317
Sherman	6503	7/3/2003	23.1	F	0.470
Sherman	6504	7/3/2003	18.6	M	0.344
Sherman	6506	7/3/2003	12.0	M	0.195
Sherman	6507	7/3/2003	17.2	M	0.258
Sherman	6509	7/3/2003	12.5	M	0.264
Sherman	6696	7/3/2003	14.6	M	0.277
Sherman	6697	7/3/2003	14.0	M	0.175
Sherman	6698	7/3/2003	22.8	F	0.443
Sherman	6699	7/3/2003	15.3	M	0.241

Sherman	6700	7/3/2003	22.4	F	0.317
Siskiwit	6637	7/9/2003	18.5	F	0.754
Siskiwit	6638	7/9/2003	18.6	F	0.759
Siskiwit	6640	7/9/2003	15.2	M	0.668
Siskiwit	6641	7/9/2003	15.1	M	0.481
Siskiwit	6642	7/9/2003	17.2	M	1.18
Siskiwit	6643	7/9/2003	15.6	F	0.490
Siskiwit	6645	7/9/2003	13.8	M	0.263
Siskiwit	6646	7/9/2003	18.1	M	1.17
Siskiwit	6647	7/9/2003	14.8	M	0.393
Siskiwit	6648	7/9/2003	17.5	M	0.673
Siskiwit	6649	7/9/2003	13.5	M	0.374
Siskiwit	6650	7/9/2003	14.8	M	0.510
Squirrel	6602	7/15/2003	14.9	M	0.193
Squirrel	6603	7/15/2003	13.3	M	0.241
Squirrel	6604	7/15/2003	19.0	F	0.253
Squirrel	6605	7/15/2003	11.0	M	0.158
Squirrel	6791	7/15/2003	24.9	F	0.716
Squirrel	6793	7/15/2003	18.6	F	0.517
Squirrel	6794	7/15/2003	20.2	F	0.648
Squirrel	6795	7/15/2003	22.3	F	0.686
Squirrel	6796	7/15/2003	16.0	F	0.260
Squirrel	6797	7/15/2003	17.1	M	0.368
Squirrel	6798	7/15/2003	24.4	F	0.451
Squirrel	6799	7/15/2003	17.5	M	0.553
Squirrel	6800	7/15/2003	12.9	M	0.172
Turtle Flambeau Fl	6703	7/29/2003	12.2	M	0.198
Turtle Flambeau Fl	6704	7/29/2003	15.2	M	0.597

Turtle Flambeau Fl	6706	7/29/2003	18.2	F	0.796
Turtle Flambeau Fl	6708	7/29/2003	15.8	M	0.539
Turtle Flambeau Fl	6710	7/29/2003	13.0	M	0.589
Turtle Flambeau Fl	6711	7/29/2003	14.9	M	0.530
Turtle Flambeau Fl	6715	7/29/2003	16.6	M	1.07
Twin (N. Twin)	6776	7/3/2003	13.9	M	0.120
Twin (N. Twin)	6777	7/3/2003	19.5	F	0.274
Twin (N. Twin)	6779	7/3/2003	18.3	F	0.210
Twin (N. Twin)	6780	7/3/2003	25.7	F	0.409
Twin (N. Twin)	6781	7/3/2003	17.8	M	0.300
Twin (N. Twin)	6782	7/3/2003	28.1	F	1.01
Twin (N. Twin)	6783	7/3/2003	17.2	M	0.277
Twin (N. Twin)	6784	7/3/2003	19.8	M	0.276
Twin (N. Twin)	6785	7/3/2003	15.2	F	0.142
Twin (N. Twin)	6786	7/3/2003	14.4	M	0.146
Twin (N. Twin)	6789	7/3/2003	26.5	F	0.733
Twin (N. Twin)	6790	7/3/2003	12.7	M	0.137
Willow Fl	6747	7/15/2003	14.9	M	0.411
Willow Fl	6748	7/15/2003	22.7	F	1.21
Willow Fl	6749	7/15/2003	17.0	F	0.516
Willow Fl	6750	7/15/2003	16.2	M	0.566
Willow Fl	6752	7/15/2003	13.3	M	0.294
Willow Fl	6753	7/15/2003	18.5	M	0.708
Willow Fl	6755	7/15/2003	18.5	F	0.516
Willow Fl	6756	7/15/2003	22.3	F	1.35
Willow Fl	6757	7/15/2003	19.2	F	0.563
Willow Fl	6758	7/15/2003	13.4	M	0.395
Willow Fl	6759	7/15/2003	19.5	F	0.994

Muskellunge

Big St.Germain	322	8/12/2003	41.0	U	0.393
Big St.Germain	323	8/12/2003	36.0	U	0.603
Chippewa	357	8/12/2003	34.2	U	0.274
Chippewa	360	8/12/2003	46.0	U	0.923
Lost Land	361	8/12/2003	34.0	F	0.247
Lost Land	366	8/12/2003	40.0	F	0.648
Minocqua	301	8/12/2003	47.5	F	1.12
Minocqua	305	8/12/2003	43.0	F	0.978
Minocqua	306	8/12/2003	34.0	M	0.437
North Twin	337	8/12/2003	42.7	F	0.444
North Twin	338	8/12/2003	37.5	M	0.547
North Twin	339	8/12/2003	37.2	M	0.612
Round Lake	367	8/12/2003	40.9	F	0.398
Round Lake	368	8/12/2003	35.4	F	0.200
Round Lake	371	8/12/2003	44.2	F	0.383
Whitefish	373	8/12/2003	34.3	M	0.378
Yellow	343	8/12/2003	44.0	F	0.728

Table 6. Percent Moisture, after Grinding, in Walleye and Muskellunge Filet Muscle Captured during the Spring of 2003.

Lake	Sample ID	% Moisture
	Walleye	
Annabelle	6621	78.96
Annabelle	6621 dup	79.11
Annabelle	6622	82.00
Annabelle	6625	81.66
Annabelle	6627	80.25
Annabelle	6632	83.19
Bass-Patterson	6682	79.03
Bass-Patterson	6683	76.83
Bass-Patterson	6684	78.72
Bass-Patterson	6685	79.04
Bass-Patterson	6685 dup	79.04
Bass-Patterson	6686	78.88
Butternut	6666	77.69
Butternut	6666 dup	77.79
Butternut	6670	78.49
Butternut	6671	78.26
Butternut	6673	78.66
Butternut	6679	79.53
Chippewa Flowage	6717	79.23
Chippewa Flowage	6717 dup	79.20
Chippewa Flowage	6720	77.68
Chippewa Flowage	6723	78.75
Chippewa Flowage	6725	79.64

Chippewa Flowage	6726	79.51
Gile Flowage	6511	80.02
Gile Flowage	6514	79.45
Gile Flowage	6518	80.56
Gile Flowage	6520	81.80
Gile Flowage	6525	80.98
Kentuck	6652	79.46
Kentuck	6653	79.43
Kentuck	6654	79.61
Kentuck	6655	78.87
Kentuck	6660	78.77
Minocqua	6763	79.43
Minocqua	6768	79.04
Minocqua	6771	79.90
Minocqua	6771 dup	79.66
Minocqua	6773	79.62
Minocqua	6774	78.60
Namekagon	3743	79.79
Namekagon	6732	80.63
Namekagon	6736	79.95
Namekagon	6739	77.93
Namekagon	6741	79.54
North Twin	6776	78.76
North Twin	6777	81.00
North Twin	6779	81.00
North Twin	6780	78.47
North Twin	6781	79.57
Owen	6527	78.61

Owen	6530	79.33
Owen	6535	77.98
Owen	6536	77.89
Owen	6539	78.73
Pike Lake (MI)	6545	78.41
Pike Lake (MI)	6550	77.73
Pike Lake (MI)	6664	78.87
Pike Lake (MI)	6678	78.26
Sherman	6506	80.59
Sherman	6507	80.28
Sherman	6509	77.99
Sherman	6696	79.81
Sherman	6696 dup	79.72
Sherman	6700	80.40
Siskiwit	6637	79.54
Siskiwit	6638	79.90
Siskiwit	6640	79.54
Siskiwit	6641	79.27
Siskiwit	6641 dup	79.23
Siskiwit	6642	79.73
Squirrel	6602	79.27
Squirrel	6603	79.32
Squirrel	6604	79.42
Squirrel	6604 dup	79.51
Squirrel	6794	78.65
Squirrel	6796	79.97
Turtle Flambeau Flowage	6704	79.02
Turtle Flambeau Flowage	6708	79.85

Turtle Flambeau Flowage	6708dup	79.62
Turtle Flambeau Flowage	6710	79.41
Turtle Flambeau Flowage	6711	80.19
Turtle Flambeau Flowage	6715	79.55
Willow Flowage	6747	78.23
Willow Flowage	6749	78.75
Willow Flowage	6750	78.74
Willow Flowage	6758	78.69
Willow Flowage	6759	79.85
	Mean	79.35
	Std. Dev.	1.02
	Muskellunge	
Big St. Germain	0322	73.33
Big St. Germain	0322 dup	73.30
Big St. Germain	0323	73.00
Chippewa	0360	76.59
Chippewa	0360 dup	76.60
Chippewa	0357	76.83
Lost Land	0361	79.31
Lost Land	0366	76.81
Minocqua	0301	77.33
Minocqua	0305	76.58
Minocqua	0305 dup	76.55
Minocqua	0306	76.95
North Twin	0337	75.93
North Twin	0338	75.01
North Twin	0339	75.12

Round Lake	0367	76.21
Round Lake	0367 dup	76.60
Round Lake	0368	79.02
Round Lake	0368 dup	79.06
Round Lake	0371	75.89
Whitefish	0373	75.71
Yellow	0343	75.78
Yellow	0343 dup	75.76
	Mean	76.23
	Std.Dev.	1.64

APPENDIX A

PROCEDURES FOR COLLECTING, PREPARING AND TRANSPORTING FISH SAMPLES

INTRODUCTION

This SOP includes general guidelines for the collection of fish samples at the study sites, preparing the specimens as samples, wrapping and labeling samples, preservation, and transportation to the laboratory for further studies. Species of fish collected may vary, and the preparation of each species may vary slightly, depending on the needs for the analysis to be performed. The objective of this SOP is to provide to the analytical laboratory samples of fish tissue that is properly identified, labeled, wrapped, preserved, and comparable from one sample to the next.

EQUIPMENT LIST

- Permanent Ink Marker
- Solvent Rinsed Aluminum Foil
- Gallon-Size Freezer Bags
- Knives Sufficient to Filet Fish
- Freezer Space for Storage of Samples
- Coolers for Shipment
- Ice for Coolers
- Log Sheet to Record Data
- Label Tape
- Pencil

PROCEDURE

1. Collect fish samples in a manner appropriate for the study.
2. Identify the species of fish for sampling.
3. Prepare a waterproof label to identify each sample (use pencils or indelible ink only).
   a. Label the species.
   b. Label the date of capture.
   c. Label the place (lake) of capture.
   d. Total length and weight of whole fish.
   e. Sex of fish (when necessary or possible).
   f. Other data as required.
4. Prepare the fish as a sample (i.e., whole animal, entrails removed, filet with skin or without skin, etc.).
5. Place sample in acetone- or hexane-rinsed aluminum foil if the sample is to be analyzed for organic materials. Place sample in a plastic bag if the sample is to be analyzed for metals.
6. Dual labels are recommended. Place a waterproof label in the package with the sample and another label on the outside of the package.
7. Place the sample on ice in the field as soon as possible (within two hours) and deliver to a freezer within the same 24-hour period.
8. Record on a separate log (sheet of paper or log book) the data that was included on the labels with the fish samples.
9. Transport sample to the laboratory in frozen condition (do not let samples thaw until ready for analysis).

Example of Label

Name of Study:	Date:
Species:	Location of Capture:
Total Length (units):	Weight (units):
Sex:	Name of Investigator:
Other Information:	

APPENDIX B

STANDARD OPERATING PROCEDURE

COLD VAPOR MERCURY ANALYSIS - ROUTINE LABWARE CLEANING

INTRODUCTION

This cleaning procedure is used for the routine cleaning of labware being used during any cold vapor mercury analysis procedures. The proper safety equipment must be worn during the entire cleaning procedure. This includes gloves, goggles, and lab coat.

EQUIPMENT LIST

- Deionized Water
- Gloves
- Lab Coat
- Micro or Liquinox Detergent
- Various Labware Washing Brushes
- Plastic Dish Rack
- Plastic 14"x10"x10" HPDE tank with cover
- Ammonium Hydroxide, 30% (reagent grade)
- Nitric Acid, Concentrated (Reagent grade)
- Dish Pan
- Goggles
- Labware to be Washed
- pH Indicator Strips
- Wash Bottle

PROCEDURE: LABWARE CLEANING

1. Scrub the labware thoroughly in hot water containing Micro or Liquinox detergent.
2. Rinse the labware with hot water until there is no presence of soap.
3. Rinse the labware once with deionized water.
4. Place the labware in the plastic tank containing 10% nitric acid. Be sure the labware is completely filled with acid. Allow the labware to soak for a minimum of 60 minutes.
5. Remove the labware from the tank, emptying the acid back into the tank.
6. Rinse the labware three times with deionized water.
7. Place the clean labware in a plastic rack to air dry. When the labware is dry, cover the labware with a lid, stopper, or aluminum foil. Place the labware in a proper storage location until used.

PROCEDURE: PLASTIC TANK CONTAINING 10% (V/V) NITRIC ACID

1. Fill the tank with 14.4 liters of deionized water. Then add 1.6 liters of concentrated nitric acid and stir. The tank is now ready to be used to soak labware.
2. Every few months change the acid in the tank. Neutralize the acid with ammonium hydroxide until a pH of between 6 and 10 is achieved. Measure the pH in the tank with pH indicator strips.
3. Pour the neutralized acid down the drain with running cold water. Run the cold water for an additional 10 minutes.
4. Rinse the tank with warm tap water and then with deionized water. Fill the tank with 10% nitric acid as in step 1.

APPENDIX C

STANDARD OPERATING PROCEDURE

COLD VAPOR MERCURY ANALYSIS - MEAT GRINDER CLEANING

INTRODUCTION

This cleaning procedure is only required for meat grinder and labware being used for grinding of fish samples for cold vapor mercury analysis. The proper safety equipment must be worn during the entire cleaning procedure. This includes gloves, goggles, and lab coat.

EQUIPMENT LIST

- Plastic Pan
- Dish Pan
- Goggles
- Liquinox Detergent
- Various Labware Washing Brushes
- Meat Grinder
- Ammonium Hydroxide, 30% (Reagent grade)
- Hydrochloric Acid, Concentrated (Reagent grade)
- Deionized Water
- Gloves
- Lab Coat
- pH Indicator Strips
- Wash Bottle
- Labware to be Washed

PROCEDURE: MEAT GRINDER AND LABWARE CLEANING

1. Dismantle the meat grinder before washing.
2. Scrub the meat grinder components and labware thoroughly in hot water containing Liquinox detergent.
3. Rinse the meat grinder components and labware with hot water until there is no presence of soap.
4. Rinse the meat grinder components and labware with deionized water.
5. Place the meat grinder components and labware in a plastic pan containing 0.1 M HCl. Be sure that the meat grinder components and labware are completely immersed in the acid. Allow the meat grinder components and labware to soak for 30 seconds.
6. Rinse the meat grinder components and labware with deionized water.
7. Assemble the meat grinder which is ready to be used.

PROCEDURE: PLASTIC PAN CONTAINING 0.1 M HYDROCHLORIC ACID

1. Fill the plastic pan with 4 liters of deionized water. Then add 33 mL of concentrated hydrochloric acid and stir. The pan is now ready to be used to soak.
2. Periodically change the acid in the plastic pan. Neutralize the acid with ammonium hydroxide until a pH of between 6 and 10 is achieved. Measure the pH in the plastic pan with pH indicator sticks.
3. Pour the neutralized waste down the drain with running cold water. Run the cold water for an additional five minutes.
4. Rinse the plastic pan with warm tap water and then with deionized water. Fill the plastic pan with 0.1 M hydrochloric acid as in step 1.

APPENDIX D

STANDARD OPERATING PROCEDURE

COLD VAPOR MERCURY ANALYSIS - FISH GRINDING

INTRODUCTION

This procedure is for the grinding of fish filets into homogeneous samples. The meat grinder and labware used to grind the fish is cleaned by the "Cold Vapor Mercury Analysis - Meat Grinder Cleaning (SA/9)" procedure. The jars the ground fish samples are placed in are cleaned by the "Cold Vapor Mercury Analysis - New Labware Cleaning (SA/15)" procedure. The proper safety equipment must be worn during the entire grinding procedure. This includes gloves, goggles, and lab coat.

EQUIPMENT LIST

- Fish Filets Samples
- Gloves
- Lab Coat
- Spatula
- Aluminum Foil
- Tuna fish
- Food Processor with Grinding Attachments
- Filet Knife
- Goggles
- Grinder
- Beaker
- Scintillation Vials

PROCEDURE: GRINDING FISH Filet SAMPLES

1. Cut the fish filets into small pieces that will fit through the grinder feed tube or food processor with grinding attachments.
2. Pass the fish through the grinder or food processor, discarding the first few grams of tissue that come through. Collect the fish tissue in a beaker.
3. Mix the fish tissue with a spatula.
4. Repeat steps 2 and 3 an additional two times.
5. Place the fish in a previously acid-cleaned container. Seal securely with the screw top lid. Label the vial with the appropriate information and place in a freezer until analyzed.
6. Wash the grinder (or food processor) and labware by the "Cold Vapor Mercury Analysis - Meat Grinder Cleaning " procedure before grinding the next fish sample.
7. Continue to grind each fish sample by steps 1 - 7.

PROCEDURE: PREPARING THE PROCEDURAL BLANK

1. Drain a can of tuna fish to be used as the procedural blank. Grind half the tuna fish as a procedural blank by use of steps 2 - 7. Label the tuna fish as "ground" and include with the analysis set.
2. The other half of the tuna is left unground and handled like a sample by use of steps 5 + 6. Label the tuna fish as "unground" and include with the analysis set.

APPENDIX E

COLD VAPOR MERCURY ANALYSIS - FISH SAMPLE WEIGHING

INTRODUCTION

This procedure is for the weighing of ground fish tissue for cold vapor mercury analysis. The fish should be ground by use of the "Cold Vapor Mercury Analysis - Fish Grinding" procedure. The labware used in this procedure should be cleaned by the "Cold Vapor Mercury Analysis - Routine Labware Cleaning" procedure. The proper safety equipment must be worn during this entire procedure. This includes gloves, safety glasses or goggles, and lab coat.

EQUIPMENT LIST

- Ground Fish Samples
- Goggles or Safety Glasses
- Nitric Acid (10%)
- Glass Bottles with Ground Glass Stoppers
- Balance Capable of Reading to the Nearest 0.001 g
- Gloves
- Lab Coat
- Spatula
- Kimwipes

PROCEDURE

1. Remove the fish to be analyzed from the freezer and allow to partially thaw.
2. Check the level of the balance and adjust if necessary. Clean the top of the balance of any foreign materials with a soft brush.
3. Zero the balance with the zero adjustment to read 0.000 g.
4. Place a clean glass bottle on the balance and measure weight. Tare the balance.
5. Weigh approximately 0.2 g - 0.3 g of fish tissue into the glass bottle.
6. Weigh and record the total weight of the glass bottle and fish tissue.
7. Rinse the spatula with water, 10% nitric acid and deionized water. Wipe the spatula clean with a Kimwipe.
8. Label and record each glass bottle and fish sample. Be sure that none of the fish tissue adheres to the side of the glass bottle.

APPENDIX F

FIMS MERCURY ANALYSIS - STOCK, STANDARD AND SPIKE PREPARATION

INTRODUCTION

This procedure is used for the preparation of the stock, analytical standards, blanks and spikes for analysis using the Perkin Elmer FIMS-100 Mercury Analyzer. The fish/tissue used for the spikes should be weighed by the use of the "Sample Weighing for Metals Analysis (SA/11)" procedure. The labware used in this procedure should be cleaned by the "Routine Labware Cleaning for Metals Analysis" (SA/8) procedure.

EQUIPMENT LIST

- Ground Tissue Samples for Spikes
- Class A Pipettes (1 mL and 3 mL)
- Deionized Water
- Pipette Bulb
- 1000 mg/L Mercuric Nitrate Stock/Reference Solution
- Concentrated Hydrochloric Acid (Trace Metal Grade)
- 5% (w/v) Potassium Permanganate ($KMnO_4$)
- Micropipettes and Tips
- Teflon Beakers for Making Substocks
- Mercury Waste Container
- 2 Volumetric Flasks (100 mL)
- Polypropylene Digestion Cups (Environmental Express)

PROCEDURE

1. Pipet 1 mL of a 1000 mg/L mercuric nitrate stock solution into a 100 mL volumetric flask containing ~60 mL of deionized water, 1 mL trace metal grade concentrated HCl, and 100 µL 5% $KMnO_4$. Dilute to 100 mL with deionized water to prepare a 10 mg/L Hg substock. Label this solution with the concentration, date and initials as it must be remade once a month.
2. Pipet 1 mL of the 10 mg/L Hg substock solution into a 100 mL volumetric flask containing ~60 mL of deionized water, 0.5 mL trace metal grade concentrated HCl, and 100 µL 5% $KMnO_4$. Dilute to 100 mL with deionzed water to prepare a 100 µg/L Hg substock. Label this solution with the concentration, date and initials as it must be remade once a week.
3. Pipet the following volumes of deionized water and 100 µg/L Hg substock into digestion cups labeled with the appropriate concentrations which are based on the final volume (50 mL) of standard at time of analysis. Use a micropipette to deliver all water volumes and stock Hg volumes less than 1 mL. Use a class A pipet to deliver 3 mL 100 µg Hg/L substock.

Concentration (ng/L)	Amount of 100 µg/L substock	Amount of DI water
Blank	0	3 mL
50	25 µL	2975 µL
100	50 µL	2950 µL
500	250 µL	2750 µL

1000	500 µL	2500 µL
6000	3 mL	0 mL

4. Each blank and standard should be prepared in duplicate.
5. A total of 10% of samples analyzed for mercury should be spiked in duplicate. Spiking is accomplished by pipetting a known volume of the 100 µg/L Hg substock into a digestion cup containing a known weight of fish tissue. A micropipette may be used to deliver two 750 µL aliquots onto pre-weighed tissue to give a total spiking volume of 1.5 mL.
6. All mercury waste from rinsing pipettes, beakers, etc. should be disposed of in mercury waste container. Volume and concentration placed in waste container should be recorded on the hazardous waste container inventory form for that bottle.

APPENDIX G

COLD VAPOR MERCURY DETERMINATION IN BIOTA

INTRODUCTION

This procedure is used for the determination of total mercury in fish, hair and other tissue samples. Do not use this procedure for analyzing human blood.

REFERENCES

"Determination of Mercury in Tissues by Cold Vapor Atomic Absorption Spectrometry", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, April 1991.

EQUIPMENT LIST

- Stannous Chloride, Analytical Reagent
- Magnesium Perchlorate, Anhydrous for Elemental Analysis
- Potassium Persulfate, Reagent Suitable for Mercury Determination
- Hydroxylamine Hydrochloride, Reagent Suitable for Mercury Determination
- Potassium Permanganate, Certified A.C.S.
- Sodium Chloride, Certified A.C.S.
- Sulfuric Acid, A.C.S. Reagent, Suitable for Mercury Determination
- Hydrochloric Acid, Trace Metals Grade
- Nitric Acid, Fisher, Trace Metals Grade
- Mercury Cold Vapor Analyzer
- Hollow Cathode Mercury Lamp
- Variable Autotransformer
- Neptune Dyna-Pump Model 4K
- Hot Block (Environmental Express)
- Varian SpectrAA 200 Spectrophotometer
- FIMS-100 (Perkin Elmer) Mercury Analyzer
- Labindustries Repipet II Dispenser, 3 - 10 mL and 1 - 5 mL
- Wheaton Instruments Socorex Dispenser Model 511, 10 mL
- Polypropylene Digestion Cups and Covers
- Pipets/Pipettors
- Beakers
- Spatulas
- 5% (w/v) Potassium Permanganate
- 5% (w/v) Potassium Persulfate
- 10% (w/v) Hydroxylamine Hydrochloride-10%(w/v) Sodium Chloride
- 10% (w/v) Stannous Chloride-0.5M Sulfuric Acid for Spectra AA Analysis
- 0.05M Potassium Permanganate-5% (v/v) Sulfuric Acid
- 1000 ug/mL Mercuric Nitrate Stock
- 5 ug/mL Mercuric Nitrate Substock for Spectra AA Analysis
- 50 ng/mL Mercuric Nitrate Substock for Spectra AA Analysis
- 10 mg/L Mercuric Nitrate Substock for FIMS-100 Analysis
- 100 ug/L Mercuric Nitrate Substock for FIMS-100 Analysis
- Silicon Defoaming Agent (Perkin Elmer)
- Deionized Water in Teflon Squirt Bottle

PROCEDURE

Digestion

1. Add 4.0 mL of concentrated sulfuric acid and 1.0 mL of concentrated nitric acid to each sample, standard, spike, duplicate and blank.
2. Place the digestion cups in Hot Block at 110°C and allow to digest for approximately 15 minutes or until all the fish tissue is dissolved.
3. Turn off the Hot Block and allow the digestion cups to cool to room temperature.
4. Add 5.0 mL of 5% potassium permanganate to each bottle in 1.0 mL increments swirling the digestion cups after each addition.
5. Add 10.0 mL of 5% potassium permanganate to each digestion cup in 5.0 mL increments, swirling the digestion cup after each addition. Additional 5% potassium permanganate solution (maximum of 5 mL) or solid potassium permanganate should be added to the samples if necessary so that the samples remain purple in color for at least 15 minutes. If extra potassium permanganate is added to a sample, an equal amount should be added to one set of standards and a blank.
6. Add 8 mL of 5% potassium persulfate to each digestion cup, and cover and swirl.
7. Allow the digestion cup to set overnight to oxidize organic mercury compounds to inorganic mercury ions.
8. The samples will remain stable for several days before analysis.

Sample Analysis Using Varian SpectraAA 200

Instrument Conditions

Current = 3.0 mA
Atomic Absorption Mode (AA)
Statistics = 99
D_2 Background Correction with diffraction grating filter
Circulating Pump autotransformer = 70% power

Wavelength = 253.7 nm
Double Beam Mode (DB)
Integration = 1.0 seconds

Instrument Conditions for Varian SpectrAA 200

Sampling Mode = AutoMix
Calibration Mode = Scale Expansion
Measurement Mode = Integrate
Replicates Standard = 20
Replicates Sample = 20
Expansion Factor 1.0
Minimum Reading = Disabled
Smoothing = 9 pt
Conc. Units = ng
Conc. Decimal places = 2

Wavelength = 253.7 nm
Slit Width = 1.0 nm
Lamp Current = 3.0 mA
Background Correction = BC on
Cal. Zero Rate = 0
Measurement Time = 4.5 s
Pre-Read Delay = 0 s
Vapor Type = Cold Vapor
Burner Height = 16.0 mm

1. Set the AA to the instrument conditions listed above and allow instrument warm-up time. Prepare the 10% stannous chloride/0.5 M sulfuric acid solution and the magnesium perchlorate drying tube. Attach the drying tube in the cold vapor mercury analyzer.
2. Autozero the AA by aerating deionized water through the cold vapor mercury analyzer.
3. Transfer the sample from the digestion cup to a glass bottle. Add 10 mL of hydroxylamine hydrochloride/10% sodium chloride to the digestion cup, then transfer to the glass bottle with the sample. Swirl sample until no purple or brown color remains. Rinse the digestion cup with three portions of deionized water, adding the rinse to the sample in the glass bottle each time. Be careful not to end up with the bottle more than two-thirds full.

4. Add 5.0 mL of 10% stannous chloride/0.5 M sulfuric acid to a sample and immediately attach to the mercury analyzer.
5. Measure the absorbance of the sample until the maximum absorbance is reached and begins to decline and record the maximum absorbance as the response.
6. Change the valves of the mercury analyzer to draw the mercury into a 0.05 M potassium permanganate/5% sulfuric acid trap. Purge the mercury analyzer of mercury until the absorbance reaches a minimum similar to the background absorbance.
7. Return the valves to the "analyze" position and rinse the aerator with deionized water before analyzing the next sample. Dispose of the analyzed and purged sample into an Acid Waste container.
8. Alternate analyzing the samples, standards and blanks by use of steps 3-7.
9. Neutralize the "Acid Waste" in a fume hood with ammonium hydroxide until the pH is between 6 and 10. Pour the neutralized waste down the drain with running cold water. Record the volume of waste neutralized in the Acid/Base Waste Log.
10. Collect the exhausted stocks and standards in a glass bottle identified as "Hazardous Waste - Mercuric Nitrate in % acid solutions. Corrosive Toxic." Note the start date. Each waste bottle will require an analysis before it will be accepted for disposal.

Sample Analysis Using Perkin Elmer FIMS-100 Flow Injection Mercury Analysis System

- Prepare the following:
  Carrier Solution (3% HCl)
  Reductant Solution (5% $SnCl_2$, 1% Silicon Defoaming Agent, in 3% HCl)
  Weigh 50g $SnCl_2$ and add to 990 mL 3% HCl. Add 10 mL Silicon Defoaming Agent using 5 mL micropipettor.
- Turn on computer and printer.
- Turn on Nitrogen (400 psi).
- Turn on FIMS 100 mercury analyzer and allow to warm up for 10 minutes minimum.
- Press Ctrl+Alt+Del (on computer).
- Username: administrator.
- Leave password field blank. Click on "OK".
- Open appropriate project Excel file prepared from Hg Calculations-Master and minimize the Excel window.
- Double click on AA Winlab Analyst icon.
- Choose "Use a custom designed workspace".
- Choose "Hg.fms" > "file" > "open" > "method" > "Hg Analysis".
- Click on "Browse" in Results Data Set window and enter a new data set name (DateProject). Be sure that the save data and print log boxes are both checked.
- Turn clamps on the peristaltic pump rollers in order to allow pump to work.
- Check filter compartment cover to see that it has been tightened.
- Attach tubing from filter compartment to cell.
- Click on Manual button (on top toolbar).
- Click on FIAS button (on top toolbar). Run FIAS once using clean deionized water (Click on the "FIAS on/off" button). Place collection tubes into appropriate solution bottles (Red = Reductant solution, Yellow = Carrier Solution) and run FIAS two more times checking the flow of the instrument and the lines for bubbles while it is running. Remember while running a sample set to periodically check carrier and reductant volumes, so they do not deplete.
- Just prior to analysis of all blanks, standards and samples (steps 19-22), add 10 mL of 10% (w/v) Hydroxylamine Hydrochloride - 10% (w/v) Sodium Chloride in two 5 mL aliquots, mix sample until no purple or brown color remains. Dilute to 50 mL with deionized water using the correct line on the digestion cup.
- Rinse the collection tube with deionized water and place in the blank solution. Click on "analyze blank" and allow instrument time to complete triplicate analysis.

- Rinse the collection tube with deionized water and place in the lowest standard. Choose appropriate standard concentration and click on "analyze standard" and allow instrument time to complete triplicate analysis. In the appropriate Excel file for that project, enter 0.000 for the blank absorbance and enter the mean Blank Corrected Signal value for the standard. Repeat this step for each of the five standards to be run in order of lowest to highest to develop the standard curve.
- Rinse the collection tube with deionized water and place in appropriate sample. Enter sample ID code into the appropriate field. Rinse the collection tube with DI water and place in appropriate sample. Click on "analyze sample" and allow instrument time to complete triplicate analysis. Enter the mean Blank Corrected Signal value into the appropriate Excel file for that project. Repeat this step for each of the samples to be analyzed.
- The second Blank, second set of standards, and Dorm-2 samples should be run as they were above, sometime in between samples, to check the precision of the instrument. For example, if the sample set contains 52 samples, including duplicates and spikes, run the first set of standards (~13 samples), the Blank and the lowest standard (50 ng/L), Dorm 2-1 (1) and (2) (~13 samples), the next two standards (100 ng/L and 500 ng/L), Dorm 2-2 (1) (~13 samples), the last two standards (1000 ng/L and 6000 ng/L) and finally Dorm 2-2 (2). It is best to try to analyze the duplicates and spikes without interruption, so more or less than 13 samples may be analyzed between standards in order to keep the samples together and in order.

WHEN ANALYSIS OF ALL SAMPLES AND STANDARDS IS COMPLETE:

- Place sample collection tube, and lines from reductant and carrier solutions into beaker of deionized water.
- Flush/clean tubing with deionized water by running FIAS two times.
- Lift collection tubing out of deionized water and run FIAS one more time to allow air to pass through all tubing. When FIAS is finished running, place collection tubing back into beaker of DI water for storage.
- Raise waste lines out of liquid in waste container so liquid does not back up.
- Release the peristaltic pump rollers so that tubing is not compressed.
- Detach line from cell.
- Unscrew the filter compartment cover and, using forceps to handle filter, dry filter with a Kimwipe.
- Print report. Choose "file" > "utilities" > "reporter" . "Open Design"
  Choose "WR01 Mussel" (double-click), then double-click on the number 1 under result name and choose the data set for that day. Click "OK" > "Print Report" and close the reporter window.
- Save Excel file to floppy disk.
- Turn off FIMS instrument, computer, nitrogen, gas and printer.
- Record the date, project, analyst, number of injections, and time run in FIMS-100 usage record book located on top of instrument.

APPENDIX H

PROCEDURES FOR DETERMINING PERCENT MOISTURE IN TISSUE SAMPLES

INTRODUCTION

This SOP includes general guidelines for the analysis of tissue samples for moisture content. It is a gravimetric technique requiring careful weighing techniques.

EQUIPMENT LIST

- Analytical Balance (i.e., Mettler AG245, PB303, AB204, H34, H72 and H80)
- Aluminum Weighing Pans
- Drying Oven (60° C)
- Desiccation Container
- Spatula

PROCEDURE

1. Calibrate analytical balance using Class One weights. Label the aluminum weighing pans and dry at 60° C for 16 hours.
2. Place dried weighing pans in desiccator until cool.
3. Weigh the dried and cooled weighing pans on an analytical balance to the 0.0001 g.
4. Weigh approximately 1.0 g of thawed tissue and place in the labeled weighing pan.
5. Weigh the pan and the tissue on an analytical balance to the nearest 0.0001 g.
6. Dry pan and tissue in drying oven at 60° C for 16 hours or until constant dry weight is achieved.
7. Remove dried pans and tissue from the oven and place in desiccator until cool.
8. Weigh the pan with the tissue on an analytical balance to the nearest 0.0001 g.
9. It may be necessary to dry the pan and tissue a second time when the tissue is a large mass. Desiccate and reweigh to prove that an equilibrium dry weight has been achieved.
10. Calculations:
    Dry Aluminum Pan - Aluminum pan with wet tissue = Wet weight of tissue
    (Aluminum pan and wet tissue weight - Aluminum pan and dry tissue /
    Wet tissue weight) X 100 = Percent moisture of tissue.

Appendix 8

Lake Superior Research Institute Final Report: Total Mercury Concentrations in Muscle Tissue from Walleye and Northern Pike Captured in Minnesota, Michigan, and Wisconsin Ceded Territory Waters During Spring 2004

Total Mercury Concentrations in Muscle Tissue from Walleye and Northern Pike Captured in Minnesota, Michigan, and Wisconsin Ceded Territory Waters During Spring 2004

by

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November 30, 2004

Introduction

Skinless muscle samples from walleye (*Stizostedion vitreum*) and northern pike (*Esox lucius*) filets captured during the spring of 2004 (with the exception of ten samples collected during 2003) from waters in the 1837 and 1842 Treaty ceded territories were analyzed for total mercury (Hg) content at the University of Wisconsin-Superior's Lake Superior Research Institute (LSRI). The samples were analyzed in three sets and are reported as three separate groups. The first group consisted of one hundred and thirty-three skinless walleye filets from twelve lakes in Minnesota that were collected and analyzed as part of U.S. Environmental Protection Agency's (EPA) Science to Achieve Results (STAR) Grant Number RD83104701-0. The second group of fish consisted of three hundred and forty-two skinless walleye from thirty-two lakes in Wisconsin and Michigan collected by tribal spearers and GLIFWC Inland Fisheries assessment crews as part of EPA Grant Number 96540801-0. The last group of fish were ten skinless walleye filets and two skinless northern pike filets from six lakes in Wisconsin and Minnesota that were previously analyzed at the UW-La Crosse Mercury Laboratory in La Crosse, Wisconsin. The final group also included ten skinless walleye filets collected from Squaw Lake, Vilas County, Wisconsin in spring 2003.

Methods

At the time fish were captured, a tribal warden or biologist was present to measure the total length of each fish. The fish were tagged with a unique number (i.e., a fish identification number) and whole fish with chain-of-custody forms were transferred to the Great Lake Indian Fish and Wildlife Commission (GLIFWC) laboratory. The samples were immediately placed on ice and were frozen within 36 hours of capture. At the GLIFWC laboratory, one or two filets were removed from each fish, the skin was removed from the filet and the filet was placed into a plastic bag along with a label containing the fish identification number (Appendix A). Sex of the fish was determined during the fileting process. A dorsal fin spine was removed from each fish to determine its age. At the LSRI laboratories, the walleye were received frozen and in good condition with chain-of-custody documentation. The samples were stored in a freezer at approximately -18 C until they were removed and thawed for processing and analysis.

Before processing the fish tissues, all glassware, utensils, and grinders were cleaned according to the appropriate methods (Appendices B and C). Each day, the fish to be processed were removed from the freezer and allowed to warm to a flexible, but stiff, consistency. The skinless filet was ground three times in a grinder. A small amount of the initial tissue that passed thorough the grinder was collected and discarded (Appendix D). A sub-sample of the ground tissue was placed into a critically cleaned glass vial and frozen until mercury analysis was conducted. The grinder was disassembled after each filet was ground and the unit was washed according to the grinder cleaning procedure (Appendix C).

Fish tissues were weighed for mercury analysis following standard laboratory procedure

(Appendix E). Mercury solutions for making tissue spikes and preparing analytical standards were prepared by the procedures in Appendix F. Mercury analyses were performed using cold vapor mercury analysis techniques on a Perkin Elmer FIMS 100 mercury analysis system (Appendix G). The method has a biota detection limit of 0.00188 µg Hg/g for a tissue mass of 0.1 g.

Moisture content of tissue was measured by difference of the wet tissue weight and the dried tissue weight (Appendix H). A portion (1 to 4 g) of ground tissue was placed into a pre-dryed and pre-weighed aluminum drying pan immediately following tissue grinding. The pan and wet tissue were immediately weighed and placed into an oven (60 C) and dried for various time intervals. Drying times varied from 24 to 96 hours. Approximately 47 percent of the walleye analyzed for mercury had moisture content determined. In addition, moisture contents was measured in all northern pike filets.

Quality Assurance

Quality of analysis was monitored by four methods: Analysis of similar fish tissues (Commercial canned tuna; *Thunnus* sp.) before and after the tissue grinding process (procedural blanks) to measure laboratory bias; analysis of dogfish shark (DORM-2, *Squalus acanthias*) from the Canadian government (certified reference material from National Research Council Canada, Ottawa, Ontario, Canada) that has a certified concentration of mercury to measure analytical accuracy; duplicate analysis of fish tissue from the same filet to measure analytical precision; and analysis of tissue with known additions of mercury to determine analytical interferences. Two sets of standard solutions with known amounts (analytical standards) of mercury were analyzed with each group (maximum of 40 samples plus QA samples) of tissue samples. These analytical solutions contained 0, 50, 100, 500, 1000 and 6000 ng Hg/L. They were prepared from a purchased 1000 ± 10 ppm mercury (made with mercuric nitrate) reference standard solution (Fisher Scientific, Pittsburgh, PA).

Duplicate agreement and spike recovery values were acceptable when in the range of >83.7 percent for duplicate agreement and 54.4 to 114 percent for spike recovery. All acceptable ranges are calculated as the mean ± 2 times the standard deviation of all analyses of the appropriate samples conducted from 7/3/03 to 8/12/03 at the LSRI laboratory.

A commercial canned tuna fish (*Thunnus sp.*) sample was used as a measurement of laboratory bias on the grinding process for sample preparation. One portion of each can was transferred directly into a sample bottle after the liquid was squeezed out of the can. The second portion was ground in the same manner as the walleye filets. This check was made to ensure that no contamination or loss of mercury was occurring in the grinding process. Results were considered acceptable when the agreement was >50 percent.

An acceptable range of mercury concentrations for DORM-2 standard reference material samples was calculated for this study based upon the analyses conducted from 7/3/03 to 8/12/03 (mean ±

2 times the standard deviation of all DORM-2 analyses). The calculated acceptable range was 3.61 to 5.16 µg Hg/g.

Prior to digestion, tissues from ten percent of the fish samples were spiked, in duplicate, with a known quantity of mercury and analyzed for recovery of the spiked mercury. Spike recovery is considered acceptable when it is in the range of 54.4 to 114 percent of the expected value. This is based upon the mean ± 2 times the standard deviation of all analyses of the spiked samples conducted from 7/3/03 to 8/12/03.

Results from fish analyzed under the Science to Achieve Results (STAR) Grant (Number RD83104701-0)

Quality Assurance – Mercury analysis of the canned tuna fish from a single occasion coincident with the grinding of walleye for the STAR grant resulted in 94.1 percent agreement (Table 1).

Analysis of the dogfish shark tissue (DORM-2) standard reference material was conducted with each set of walleye tissues analyzed for a total of 11 analyses (Table 2). The certified mercury concentration for the dogfish tissue was 4.64 ± 0.26 µg Hg/g. The grand mean and standard deviation was 93.3 ± 5.33 percent of the certified value. All analyses were within the acceptance range for DORM-2 samples

Fish tissues were analyzed in duplicate 18 times. Two portions of the same tissue were digested and analyzed independently sixteen times. Relative agreement between two mercury analyses of the same tissue averaged 92.8 ± 4.60 percent (Table 3). On two occasions, duplicate agreement was below the acceptable limit. Those samples were re-analyzed on 10/13/04 and resulted in acceptable values.

Samples of tissue were spiked with known concentrations of mercury prior to digestion. Mean recovery for the 14 spiked samples was 99.8 ± 8.19 percent (Table 4). All analyses were within the acceptance range for spike recoveries.

Mercury Analysis – Skinless filets of 133 walleye from 12 lakes in Minnesota were analyzed for total mercury concentration. Total mercury concentrations on a wet weight basis (Table 5) ranged from 0.054 to 1.26 µg Hg/g (parts per million).

Tissue Moisture Analysis – Percent moisture was measured in the muscle of 56 of the 133 (41.2 %) ground filets immediately following grinding (Table 6). Walleye muscle tissue contained an average of 78.2 ± 2.89 percent moisture.

Table 1. Percent Agreement of Procedural Blank Samples [Commercial Tuna Fish (Thunnus sp.) Samples Before and After Grinding] for Total Mercury Coincident with the STAR Grant Fish Analysis.

Date of Analysis	Grinding Date	Before Grinding (µg Hg/g)	After Grinding (µg Hg/g)	Mean	Relative* Percent Agreement
6/22/2004	6/1/2004	0.229	0.243	0.236	94.1

* The absolute value of the difference between before and after grinding divided by the mean of before and after grinding, minus 1, times 100.

Table 2. Mercury Concentrations of Dogfish Tissue Supplied by the National Research Council Canada (DORM-2) Coincident with STAR Grant Fish Analysis. The Tissue has a Certified Mercury Concentration of 4.64 ± 0.26 µg Hg/g Tissue.

Date of Analysis	Sample #1	Sample #2	Mean	Std. Dev.	Percent of Expected
6/22/2004	4.79	4.57	4.72	0.16	101
6/22/2004	4.17	4.99	4.58	0.58	98.7
6/29/2004	3.85	4.02	3.94	0.11	84.8
6/29/2004	4.21	4.35	4.28	0.09	92.3
7/1/2004	4.50	4.18	4.34	0.23	93.4
7/1/2004	4.49	4.07	4.28	0.30	92.2
7/7/2004	4.93	4.20	4.56	0.51	98.4
7/7/2004	3.83	3.94	3.88	0.08	83.7
7/22/2004	4.80	3.83	4.32	0.69	93.0
7/22/2004	5.00	3.66	4.33	0.95	93.3
1/25/2005	4.44	4.37	4.41	0.05	95.0
		Mean ± Std. Dev.	4.33 ± 0.25		93.3 ± 5.33

Table 3. Relative percent Agreement Between Duplicate Analysis for Total Mercury (Wet Weight) Content in Skinless Fillet Tissue of Walleye Coincident with STAR Grant Fish Analysis.

	Sample #1	Sample #2	Relative Percent

Date of Analysis	Sample ID	(µg Hg/g)	(µg Hg/g)	Mean	Agreement
6/22/2004	Knife 08782	0.145	0.170	0.157	84.1
6/22/2004	Little Rock 00065	0.075	0.076	0.076	98.7
6/22/2004	Rush Lake East 09580	0.110	0.100	0.105	90.5
6/22/2004	Little Elk 08734	0.487	0.501	0.494	97.2
6/29/2004	South Long 8816	0.149	0.157	0.153	94.8
7/1/2004	South Long 8827	0.328	0.377	0.353	86.1
7/1/2004	South Big Pine 8767	0.340	0.319	0.329	93.6
7/1/2004	Chisago 9502	0.171	0.211	0.191	79.1*
7/1/2004	Rush Lake West 9570	0.116	0.123	0.120	94.2
7/7/2004	South Center 9533	0.168	0.168	0.168	100
7/7/2004	South Center 9539	0.275	0.246	0.261	88.9
7/7/2004	South Lindstrom 9555	0.165	0.154	0.159	93.1
7/7/2004	Green 8728	0.345	0.261	0.303	72.3*
7/22/2004	Typo 9522	0.109	0.116	0.113	93.8
10/13/2004	Chisago 9502	0.163	0.164	0.163	99.4
10/13/2004	Green 8728	0.305	0.277	0.291	90.4
1/25/2005	Little Elk 8741	0.322	0.292	0.307	90.2
1/25/2005	Little Elk 8742	0.137	0.124	0.131	90.0
			Mean ± Std. Dev.		92.8 ± 4.60

* Duplicate agreement values were below the acceptable value. Samples were re-analyzed on 10/13/04

Table 4. Percent of Mercury Recovered from Skinless Walleye Fillet Samples Spiked with a Known Quantity of Mercury Coincident with the Analysis of Walleye (2004) Coincident with STAR Grant Fish Analysis.

Date of Analysis	Sample ID	Spike #1	Spike #2	Mean	Std. Dev.
6/22/2004	Knife 08782	114	94.9	104	13.3
6/22/2004	Little Rock 00065	96.3	89.8	93.0	4.60
6/22/2004	Rush Lake East 09580	104	91.5	97.8	8.89
6/22/2004	Little Elk 08734	78.2	100	89.1	15.4
6/29/2004	South Long 8816	105	102	103	2.2
7/1/2004	South Long 8827	116	112	114	3.0
7/1/2004	South Big Pine 8767	119	82.1	100	26.0
7/1/2004	Chisago 9502	103	108	106	4.0
7/1/2004	Rush Lake West 9570	124	92.0	108	22.9
7/7/2004	South Center 9533	102	106	104	2.6
7/7/2004	South Center 9539	100	97.3	98.7	1.9
7/7/2004	South Lindstrom 9555	82.0	86.4	84.2	3.1
7/7/2004	Green 8728	83.7	93.5	88.6	6.9
7/22/2004	Typo 9522	104	102	103	1.3

			Mean ± Std. Dev.		99.8 ± 8.19

Table 5. Total Mercury Concentration (Wet Weight) in Walleye Fillets from Various Length and Sex Fish Captured in the Spring of 2004 for the STAR Grant Fish Analysis.

Lake	Sample ID	Date Analyzed	Fresh Total Length (in)	Sex	µg Hg/g
Chisago	9501	7/1/2004	14.7	F	0.195
Chisago	9502	7/1/2004	14.4	F	0.191
Chisago	9504	7/1/2004	15.0	F	0.204
Chisago	9505	7/1/2004	15.6	M	0.110
Chisago	9507	7/1/2004	28.8	F	0.720
Chisago	9508	7/1/2004	24.6	F	0.464
Chisago	9510	7/1/2004	13.9	F	0.224
Chisago	9511	7/1/2004	16.0	M	0.267
Chisago	9513	7/1/2004	25.7	M	0.643
Chisago	9514	7/1/2004	27.7	F	0.742
Chisago	9515	7/1/2004	14.5	M	0.218
Chisago	9556	7/1/2004	15.5	M	0.226
Green	8717	7/7/2004	27.9	F	0.917
Green	8718	7/7/2004	28.2	F	0.615
Green	8719	7/7/2004	21.8	M	0.355

Green	8720	7/7/2004	13.3	M	0.165
Green	8721	7/7/2004	18.4	M	0.329
Green	8722	7/7/2004	19.8	M	0.343
Green	8723	7/7/2004	24.9	M	0.509
Green	8724	7/7/2004	16.1	M	0.219
Green	8725	7/7/2004	13.3	M	0.256
Green	8726	7/7/2004	15.6	M	0.306
Green	8727	7/7/2004	13.0	M	0.192
Green	8728	7/7/2004	15.1	M	0.303
Knife	8776	6/22/2004	15.1	F	0.118
Knife	8777	6/22/2004	12.8	F	0.130
Knife	8778	6/22/2004	14.1	F	0.179
Knife	8779	6/22/2004	12.7	M	0.179
Knife	8780	6/22/2004	15.2	F	0.099
Knife	8781	6/22/2004	14.2	F	0.122
Knife	8782	6/22/2004	13.5	F	0.159
Knife	8783	6/22/2004	24.7	F	0.364
Knife	8784	6/22/2004	12.5	F	0.112
Knife	8786	6/22/2004	16.8	F	0.108
Knife	8787	6/22/2004	13.8	F	0.118
Knife	8789	6/22/2004	28.1	F	0.486

Little Elk	8731	6/22/2004	23.3	F	0.208
Little Elk	8732	6/22/2004	21.7	M	0.243
Little Elk	8734	6/22/2004	26.4	F	0.487
Little Elk	8735	6/22/2004	21.6	M	0.155
Little Elk	8736	6/22/2004	19.8	M	0.108
Little Elk	8741	6/22/2004	27.3	F	0.307**
Little Elk	8742	6/22/2004	21.2	M	0.131**
Little Rock	17	6/22/2004	18.6*	F	0.233
Little Rock	24	6/22/2004	14.1*	F	0.112
Little Rock	27	6/22/2004	12.8*	M	0.115
Little Rock	64	6/22/2004	12.1*	M	0.100
Little Rock	65	6/22/2004	12.5*	M	0.076
Little Rock	68	6/22/2004	12.3*	M	0.081
Little Rock	69	6/22/2004	12.2*	M	0.092
Rush East	9577	6/22/2004	14.3	M	0.097
Rush East	9578	6/22/2004	16.8	M	0.076
Rush East	9579	6/22/2004	17.3	M	0.167
Rush East	9580	6/22/2004	13.7	M	0.105
Rush East	9581	6/22/2004	15.7	M	0.131
Rush East	9582	6/22/2004	18.7	M	0.167
Rush East	9583	6/22/2004	13.0	M	0.152

Rush East	9584	6/22/2004	21.4	M	0.217
Rush East	9585	6/22/2004	29.1	F	0.462
Rush East	9586	6/22/2004	27.4	F	0.277
Rush East	9587	6/22/2004	18.7	M	0.162
Rush East	9588	6/29/2004	25.4	M	0.197
Rush West	9561	7/1/2004	29.8	F	0.629
Rush West	9562	7/1/2004	21.3	M	0.307
Rush West	9563	7/1/2004	19.7	M	0.257
Rush West	9564	7/1/2004	15.1	M	0.248
Rush West	9565	7/1/2004	19.8	M	0.217
Rush West	9566	7/1/2004	16.9	M	0.119
Rush West	9567	7/1/2004	25.5	M	0.360
Rush West	9568	7/1/2004	29.3	F	0.775
Rush West	9570	7/1/2004	14.7	M	0.120
Rush West	9571	7/1/2004	17.1	M	0.174
Rush West	9589	7/1/2004	13.8	M	0.066
Rush West	9590	7/1/2004	13.0	M	0.054
South Big Pine	8764	7/1/2004	12.8	M	0.393
South Big Pine	8766	7/1/2004	13.8	M	0.508
South Big Pine	8767	7/1/2004	15.2	M	0.329
South Big Pine	8770	7/1/2004	13.7	F	0.404

South Big Pine	8773	7/1/2004	21.1	M	1.15
South Big Pine	8774	7/1/2004	16.3	M	0.570
South Big Pine	8775	7/1/2004	24.0	F	1.26
South Center	9531	7/7/2004	13.6	M	0.147
South Center	9532	7/7/2004	12.9	M	0.169
South Center	9533	7/7/2004	13.7	F	0.168
South Center	9534	7/7/2004	28.2	F	0.632
South Center	9535	7/7/2004	22.6	M	0.512
South Center	9538	7/7/2004	12.3	M	0.135
South Center	9539	7/7/2004	15.6	M	0.261
South Center	9540	7/7/2004	20.2	F	0.220
South Center	9541	7/7/2004	19.4	M	0.293
South Center	9542	7/7/2004	22.3	M	0.401
South Center	9543	7/7/2004	16.7	M	0.207
South Center	9544	7/7/2004	21.9	F	0.334
South Center	9545	7/7/2004	17.6	M	0.191
South Lindstrom	9546	7/7/2004	25.4	F	0.424
South Lindstrom	9547	7/7/2004	24.2	M	0.432
South Lindstrom	9548	7/7/2004	26.6	F	0.615
South	9549	7/7/2004	12.2	M	0.152

Lindstrom					
South Lindstrom	9550	7/7/2004	20.4	M	0.269
South Lindstrom	9551	7/7/2004	16.4	M	0.240
South Lindstrom	9552	7/7/2004	20.3	M	0.271
South Lindstrom	9553	7/7/2004	21.3	M	0.356
South Lindstrom	9555	7/7/2004	14.8	M	0.159
South Lindstrom	9557	7/7/2004	13.3	F	0.147
South Lindstrom	9559	7/7/2004	15.1	M	0.198
South Lindstrom	9560	7/7/2004	16.6	M	0.249
South Long	8816	6/29/2004	12.9	M	0.153
South Long	8817	6/29/2004	15.9	M	0.183
South Long	8818	6/29/2004	14.4	M	0.175
South Long	8819	6/29/2004	23.5	F	0.456
South Long	8820	6/29/2004	14.6	M	0.160
South Long	8821	6/29/2004	20.8	M	0.330
South Long	8822	7/1/2004	21.5	M	0.441
South Long	8823	7/1/2004	19.6	M	0.280

South Long	8824	7/1/2004	23.1	F	0.194
South Long	8825	7/1/2004	14.7	M	0.141
South Long	8826	7/1/2004	23.1	F	0.197
South Long	8827	7/1/2004	16.3	M	0.353
South Long	8828	7/1/2004	21.2	F	0.612
South Long	8829	7/1/2004	13.2	M	0.157
South Long	8830	7/1/2004	16.1	M	0.251
Typo	9516	7/22/2004	14.0	M	0.172
Typo	9517	7/22/2004	16.0	M	0.172
Typo	9518	7/22/2004	17.4	M	0.276
Typo	9519	7/22/2004	14.6	F	0.158
Typo	9520	7/22/2004	14.7	M	0.167
Typo	9521	7/22/2004	14.7	M	0.133
Typo	9522	7/22/2004	16.6	M	0.113
Typo	9523	7/22/2004	15.6	F	0.218
Typo	9525	7/22/2004	14.1	M	0.164
Typo	9526	7/22/2004	13.8	M	0.113
Typo	9527	7/22/2004	14.1	M	0.142
Typo	9528	7/22/2004	15.9	M	0.29

* Frozen Length measured at the GLIFWC laboratory.

** Fish 08741 & 08742 were re-analyzed in duplicate on 1/26/05. Reported results are the mean of those duplicates.

Table 6. Percent Moisture in Walleye Fillets Measured Immediately After Grinding for the STAR Grant Fish Analysis.

Lake	Sample ID	Percent Moisture	Duplicate Agreement
Chisago	9501	79.3	
Chisago	9502	77.6	
Chisago	9507	79.8	
Chisago	9556	78.3	
Green	8720	78.1	
Green	8725	77.4	99.6
Green	08725 dup	77.1	
Green	8726	78.6	
Green	8727	77.9	
Knife	8778	79.2	99.7
Knife	8778[a]	79.4	
Knife	8780	79.9	99.9
Knife	8780[a]	80.0	
Knife	8783	79.8	100.0
Knife	8783[a]	79.9	
Knife	8787	79.2	100.0
Knife	8787[a]	79.2	
Little Elk	8732	75.6	
Little Elk	8734	79.9	99.3

Little Elk	8734 dup	79.4	
Little Elk	8736	77.9	
Little Elk	8741	79.0	
Little Rock	17	79.2	
Little Rock	64	80.8	
Little Rock	65	79.6	
Little Rock	69	80.9	
Rush East	9577	78.3	
Rush East	9578	78.3	
Rush East	9579	78.6	99.6
Rush East	9579 dup	78.3	
Rush East	9580	78.2	
Rush West	989	77.9	
Rush West	9561	79.1	
Rush West	9562	78.0	
Rush West	9563	78.9	
Rush West	9564	77.8	
Rush West	9565	78.1	
Rush West	9566	78.2	
Rush West	9567	77.8	
Rush West	9568	79.0	

Rush West	9570	77.5	
Rush West	9571	78.2	
Rush West	9590	78.4	
South Big Pine	8764	78.3	
South Big Pine	8766	79.3	
South Big Pine	8767	79.1	
South Big Pine	8770	79.7	
South Big Pine	8773	78.6	
South Big Pine	8774	79.2	
South Big Pine	8775	79.0	
South Center	9532	78.4	99.4
South Center	9532[a]	78.8	
South Center	9539	79.4	99.8
South Center	9539[a]	79.2	
South Center	9540	78.3	99.8
South Center	9540[a]	78.5	
South Center	9541	62.7	99.9
South Center	9541[a]	62.8	
South Lindstrom	9547	77.9	
South Lindstrom	9551	78.4	
South Lindstrom	9553	76.6	

South Lindstrom	9560	77.9	
South Lindstrom	9564	78.1	
Typo	9520	79.3	99.8
Typo	9520 dup	79.3	
Typo	9521	78.9	
Typo	9527	80.2	
Typo	9528	79.4	
	Mean ± Std. Dev.	78.2 ± 2.89	

[a] Indicates that sample was dried in oven for 24 hours, weighed and returned to oven for 24 more hours prior to weighing for a second time

Results from fish tissues analyzed using EPA Supplemental Funds (Number 96540801-0)

Quality Assurance – Mercury analysis of the canned tuna fish from 18 occasions coincident with the grinding of walleye collected for the GLIFWC-EPA grant resulted in a mean of 85.2 ± 11.0 percent agreement (Table 7). All percent agreement values were above the minimal acceptance value of 50 percent.

Analysis of the dogfish shark tissue (DORM-2) standard reference material was conducted with each set of walleye tissues analyzed for a total of 21 analyses (Table 8). The certified mercury concentration for the dogfish tissue was 4.64 ± 0.26 µg Hg/g. The grand mean and standard deviation was 92.3 ± 7.11 percent of the certified value.

Fish tissues were analyzed in duplicate 39 times. Two portions of the same tissue were digested and analyzed independently. Agreement between two mercury analyses of the same tissue averaged 93.5 ± 5.56 percent (Table 9). Seven of the percent agreement values were below the acceptance range and were analyzed a second time and the results of the second analysis were within the acceptance range.

Samples of tissue were spiked with known concentrations of mercury prior to digestion. Mean recovery for the 34 spiked samples was 92.4 ± 10.4 percent (Table 10). All spike recovery analyses were within the acceptance range.

Mercury Analysis – Skinless filets of 332 walleye from 33 lakes in Wisconsin were analyzed for total mercury concentration. Total mercury concentrations on a wet weight basis (Table 11)

ranged from 0.089 to 1.20 µg Hg/g (parts per million).

Tissue Moisture Analysis – Percent moisture was measured in 160 fish of the 342 fish immediately following grinding (Table 12). Walleye muscle tissue had a mean moisture value of 79.2 ± 1.19 percent moisture.

Table 7. Percent Agreement of Procedural Blank Samples [Commercial Tuna Fish (Thunnus sp.) Before and After Grinding for Total Mercury Coincident with the GLIFWC EPA Fish Analysis.

Date of Analysis	Grinding Date	Before Grinding (µg Hg/g)	After Grinding (µg Hg/g)	Relative Percent Agreement
8/4/2004	7/15/2004	0.074	0.078	94.7
8/4/2004	7/21/2004	0.304	0.275	90.0
8/11/2004	7/30/2004	0.075	0.060	77.8
8/11/2004	8/4/2004	0.078	0.074	94.7
8/17/2004	7/12/2004	0.046	0.047	97.8
8/17/2004	7/30/2004	0.076	0.077	98.7
8/18/2004	7/20/2004	0.054	0.080	61.2*
8/18/2004	7/27/2004	0.080	0.053	59.4*
8/23/2004	7/12/2004	0.041	0.046	88.5
8/23/2004	7/28/2004	0.091	0.102	88.6
8/26/2004	7/15/2004	0.071	0.058	79.8
8/26/2004	8/4/2004	0.079	0.085	92.7
8/31/2004	7/27/2004	0.083	0.071	84.4
8/31/2004	7/28/2004	0.089	0.100	88.4

9/17/2004	7/30/2004	0.067	0.055	80.3
9/17/2004	8/4/2004	0.081	0.073	89.6
10/13/2004	7/20/2004	0.076	0.061	78.1
10/13/2004	7/27/2004	0.073	0.065	88.4
			Mean ± Std. Dev.	85.2 ± 11.0

* Procedural blank samples had agreement less than the acceptable limit and were re-analyzed on 10/13/04.

Table 8. Mercury Concentrations of Dogfish Tissue Supplied by the National Research Council Canada (DORM-2) Coincident with the GLIFWC EPA Fish Analysis. The Tissue has a Certified Mercury Concentration of 4.64 ± 0.26 µg Hg/g Tissue.

Date of Analysis	Sample #1	Sample #2	Mean	Std. Dev.	Percent of Expected
7/22/2004	4.80	3.83	4.32	0.69	93.0
7/22/2004	5.00	3.66	4.33	0.95	93.3
8/4/2004	4.22	3.85	4.03	0.27	87.0
8/4/2004	4.37	3.30	3.84	0.75	82.7
8/11/2004	4.70	4.09	4.39	0.43	94.7
8/11/2004	4.01	3.97	3.99	0.03	86.1
8/17/2004	3.97	3.86	3.92	0.08	84.5
8/17/2004	4.26	5.13	4.69	0.62	101.2
8/18/2004	5.15	3.88	4.52	0.90	97.3
8/18/2004	4.08	4.06	4.07	0.02	87.7

8/23/2004	4.65	3.75	4.20	0.64	90.5
8/23/2004	3.62	4.12	3.87	0.36	83.4
8/26/2004	4.00	5.16	4.58	0.83	98.7
8/26/2004	3.70	5.19	4.44	1.05	95.8
8/31/2004	3.85	3.71	3.78	0.10	81.4
8/31/2004	4.23	4.68	4.46	0.32	96.1
9/17/2004	5.20	4.22	4.71	0.69	101.5
9/17/2004	4.17	3.88	4.03	0.21	86.8
10/13/2004	4.40	4.27	4.34	0.09	93.4
10/13/2004	5.94	4.14	5.04	1.27	108.6
1/25/2005	4.44	4.37	4.41	0.05	95.0
		Mean ± Std. Dev	4.28 ± 0.33	.	92.3 ± 7.11

Table 9. Percent Agreement Between Duplicate Analysis for Total Mercury (Wet Weight) Content in Skinless Fillet Tissue of Walleye Coincident with the GLIFWC EPA Fish Analysis.

Date of Analysis	Sample ID	Sample #1 (µg Hg/g)	Sample #2 (µg Hg/g)	Mean	Percent Relative Agreement
7/22/2004	Turtle Flambeau Flowage 10891	0.405	0.370	0.388	91.0
7/22/2004	High 10783	0.221	0.167	0.194	72.2*
7/22/2004	Alder 1846	0.407	0.424	0.416	95.9

8/4/2004	Nebagamon 10716	0.446	0.458	0.452	97.3
8/4/2004	Big Portage 11235	0.225	0.247	0.236	90.7
8/4/2004	Crab 1579	0.517	0.569	0.543	90.4
8/4/2004	Lucerne 8796	0.260	0.198	0.229	72.9*
8/11/2004	Lac Vieux Desert 10840	0.157	0.160	0.158	98.1
8/11/2004	Chippewa 10848	0.310	0.222	0.266	66.9*
8/11/2004	Balsam 11209	0.155	0.150	0.152	96.7
8/17/2004	Pelican 11288	0.136	0.124	0.130	90.8
8/17/2004	Nokomis 11223	0.282	0.284	0.283	99.3
8/17/2004	Rest 53	0.162	0.154	0.158	94.9
8/17/2004	Roberts 10818	0.387	0.399	0.393	96.9
8/18/2004	Clear 8878	0.235	0.228	0.231	97.0
8/18/2004	Spider 11211	0.372	0.369	0.371	99.2
8/18/2004	Laura 11249	0.387	0.296	0.342	73.4*
8/18/2004	Wild Rice 70	0.212	0.262	0.237	78.9*
8/23/2004	Bass-Patterson 10204	0.272	0.230	0.251	83.3
8/23/2004	Island 1832	0.418	0.396	0.407	94.6
8/23/2004	Round 10870	0.107	0.112	0.110	95.5
8/23/2004	Nelson 10791	0.227	0.191	0.209	82.8
8/26/2004	Sevenmile 10804	0.658	0.593	0.626	89.6

8/26/2004	Sherman 10226	0.253	0.187	0.220	70.0*
8/26/2004	Spider 10884	0.313	0.268	0.291	84.5
8/31/2004	Little Star 37	0.276	0.277	0.277	99.6
8/31/2004	Long 10236	0.213	0.210	0.212	98.6
8/31/2004	Middle Eau Claire 10739	0.537	0.430	0.484	77.9*
9/17/2004	Manitowish 25	0.326	0.330	0.328	98.8
9/17/2004	Upper Eau Claire 10752	0.381	0.388	0.384	98.2
9/17/2004	Gogebic 10768	0.242	0.225	0.234	92.7
9/17/2004	Upper St. Croix 10712	0.188	0.224	0.206	82.5
10/13/2004	High 10783	0.192	0.163	0.178	83.7
10/13/2004	Laura 11249	0.290	0.293	0.291	99.0
10/13/2004	Lucerne 8796	0.123	0.144	0.134	84.3
10/13/2004	Middle Eau Claire 10739	0.366	0.371	0.369	98.6
10/13/2004	Sherman 10226	0.216	0.204	0.210	94.3
10/13/2004	Spider 11211	0.403	0.435	0.419	92.4
10/13/2004	Wild Rice 70	0.310	0.300	0.305	96.7
1/25/2005	Lucerne 8800	0.385	0.397	0.391	96.9
				Mean ± Std. Dev.	93.5 ± 5.56

* Duplicate agreement was below the acceptable limit, sample re-analyzed 10/13/04.

Table 10. Percent of Mercury Recovered from Skinless Walleye Fillet Samples Spiked with a Known Quantity of Mercury Coincident with the Analysis of GLIFWC EPA Walleye (2004).

Date of Analysis	Sample ID	Spike #1	Spike #2	Mean	Std. Dev.
7/22/2004	Turtle Flambeau Flowage 10891	84.2	78.8	81.5	3.83
7/22/2004	High 10783	95.6	96	95.8	0.29
7/22/2004	Alder 1846	89.5	87.1	88.3	1.71
8/4/2004	Nebagamon 10716	76.6	94	85.3	12.3
8/4/2004	Big Portage 11235	99.7	95.8	97.7	2.79
8/4/2004	Crab 1579	86.3	124	105	26.8
8/4/2004	Lucerne 8796	93.3	94.2	93.8	0.63
8/11/2004	Lac Vieux Desert 10840	106	95.4	101	7.34
8/11/2004	Chippewa 10848	93.9	108	101	10.1
8/11/2004	Balsam 11209	84.9	96	90.5	7.87
8/17/2004	Pelican 11288	83.4	79.8	81.6	2.61
8/17/2004	Nokomis 11223	73	88.9	81	11.3
8/17/2004	Rest 53	91.5	96.3	93.9	3.45
8/17/2004	Roberts 10818	80.7	84.8	82.8	2.93
8/18/2004	Clear 8878	75.5	61.8	68.6	9.71
8/18/2004	Spider 11211	128*	106	117	16.1
8/18/2004	Laura 11249	107	95.4	101	8.13

8/18/2004	Wild Rice 70	101	110	105	6.15
8/23/2004	Bass-Patterson 10204	86.6	86	86.3	0.39
8/23/2004	Island 1832	90	77.2	83.6	9.04
8/23/2004	Round 10870	95.8	93.3	94.5	1.75
8/23/2004	Nelson 10791	90.1	89.6	89.9	0.36
8/26/2004	Sevenmile 10804	82.3	70.2	76.2	8.57
8/26/2004	Sherman 10226	102	93.8	97.8	5.7
8/26/2004	Spider 10884	91.2	92.4	91.8	0.87
8/31/2004	Little Star 37	100	101	100	0.77
8/31/2004	Long 10236	98.8	98.3	98.5	0.37
8/31/2004	Middle Eau Claire 10739	85.7	86.9	86.3	0.85
9/17/2004	Manitowish 25	94.1	95.1	94.6	0.67
9/17/2004	Upper Eau Claire 10752	103	98.2	100	3.19
9/17/2004	Gogebic 10768	87.9	91	89.5	2.18
9/17/2004	Upper St. Croix 10712	112	113	113	0.97
10/13/2004	Spider 11211	73.2	83	78.1	6.9
1/25/2005	Lucerne 8800	88.6	90.8	89.7	1.57
			Mean ± Std. Dev.	92.4 ± 10.4	

* Spike recovery below acceptable limit, sample re-analyzed 10/13/04.

Table 11. Total Mercury Concentration (Wet Weight) in Walleye Fillets from Various Length and Sex Fish Captured for GLIFWC EPA in the Spring of 2004.

Lake	Sample ID	Date Analyzed	Fresh Length (in)	Sex	µg Hg/g
Alder	2	7/22/2004	13.7*	F	0.283
Alder	3	7/22/2004	12*	M	0.295
Alder	7	7/22/2004	17.8*	F	0.209
Alder	8	7/22/2004	14.4*	M	0.391
Alder	1846	7/22/2004	19.5	F	0.497
Alder	1848	7/22/2004	16.4	M	0.411
Alder	1851	7/22/2004	16.5	M	0.416
Alder	1852	7/22/2004	23.1	F	0.417
Ballard	11263	8/23/2004	15.2	M	0.683
Balsam	11204	8/11/2004	16.5	M	0.181
Balsam	11208	8/11/2004	19.5	M	0.181
Balsam	11209	8/11/2004	15.3	M	0.152
Bass-Patterson	10201	8/23/2004	16.4	F	0.264
Bass-Patterson	10202	8/23/2004	12.7	M	0.190
Bass-Patterson	10204	8/23/2004	14.5	M	0.270
Bass-Patterson	10207	8/23/2004	18.3	F	0.379
Bass-Patterson	10208	8/23/2004	25.2	F	0.510
Bass-Patterson	10210	8/23/2004	19.8	F	0.363
Bass-Patterson	10211	8/23/2004	17.8	F	0.256
Bass-Patterson	10212	8/23/2004	17.9	F	0.265
Bass-Patterson	10213	8/23/2004	18.2	M	0.331
Bass-Patterson	10215	8/23/2004	13.7	M	0.153
Big Portage	11231	8/4/2004	18.0	M	0.271
Big Portage	11232	8/4/2004	16.6	M	0.661

Big Portage	11233	8/4/2004	14.0	M	0.220
Big Portage	11235	8/4/2004	14.5	M	0.236
Big Portage	11236	8/4/2004	14.3	M	0.156
Big Portage	11244	8/4/2004	17.2	M	0.263
Big Portage	11245	8/4/2004	15.5	M	0.305
Chippewa	10846	8/11/2004	12.6	M	0.174
Chippewa	10847	8/11/2004	16.6	M	0.365
Chippewa	10848	8/11/2004	14.8	M	0.266
Chippewa	10850	8/11/2004	18.6	M	0.657
Chippewa	10852	8/11/2004	17.4	M	0.504
Chippewa	10853	8/11/2004	24.3	F	0.344
Chippewa	10854	8/11/2004	21.4	M	0.343
Chippewa	10855	8/11/2004	23.6	F	0.909
Chippewa	10856	8/11/2004	16.6	M	0.378
Chippewa	10858	8/11/2004	18.2	M	0.625
Chippewa	10859	8/11/2004	22.9	F	0.456
Chippewa	10860	8/11/2004	12.5	M	0.311
Clear	8876	8/18/2004	12.5*	M	0.157
Clear	8878	8/18/2004	17.5	M	0.231
Clear	8881	8/18/2004	18.7*	F	0.318
Clear	8883	8/18/2004	17.7*	M	0.226
Clear	8885	8/18/2004	13.2*	M	0.143
Clear	8887	8/18/2004	17.9*	M	0.148
Crab	1571	8/4/2004	13.2	M	0.369
Crab	1572	8/4/2004	19.3	F	0.564
Crab	1573	8/4/2004	12.7	M	0.562
Crab	1575	8/4/2004	16.1	F	0.601
Crab	1577	8/4/2004	15.3	F	0.542
Crab	1578	8/4/2004	18.5	F	0.719

Crab	1579	8/4/2004	14.2	M	0.543
Crab	1580	8/4/2004	17.2	M	0.763
Crab	1583	8/4/2004	23.3	F	0.659
Gogebic	10761	9/17/2004	18.7	M	0.328
Gogebic	10762	9/17/2004	22.6	F	1.01
Gogebic	10763	9/17/2004	18.4	M	0.287
Gogebic	10764	9/17/2004	18.8	M	0.507
Gogebic	10765	9/17/2004	13.5	M	0.199
Gogebic	10767	9/17/2004	18.0	M	0.356
Gogebic	10768	9/17/2004	14.0	M	0.234
Gogebic	10770	9/17/2004	23.5	F	0.485
Gogebic	10771	9/17/2004	19.3	M	0.317
Gogebic	10772	9/17/2004	18.4	M	0.434
Gogebic	10773	9/17/2004	13.2	M	0.277
Gogebic	10775	9/17/2004	23.7	F	0.809
High	10777	7/22/2004	12.0	M	0.141
High	10778	7/22/2004	16.1	M	0.250
High	10779	7/22/2004	24.2	F	0.931
High	10780	7/22/2004	15.8	M	0.263
High	10781	7/22/2004	20.2	F	0.230
High	10782	7/22/2004	18.7	F	0.255
High	10783	7/22/2004	16.5	M	0.194
High	10784	7/22/2004	20.4	F	0.709
High	10785	7/22/2004	22.6	F	0.486
High	10787	7/22/2004	12.7	M	0.127
High	10789	7/22/2004	23.4	F	0.747
High	10790	7/22/2004	13.0	M	0.154
Island	91	8/23/2004	13.9	M	0.290
Island	93	8/23/2004	17.0	F	0.402

Island	95	8/23/2004	16.2	M	0.430
Island	97	8/23/2004	14.8	M	0.487
Island	1832	8/23/2004	15.7	M	0.407
Island	1834	8/23/2004	19.5	F	0.432
Island	1835	8/23/2004	19.7	F	0.589
Island	8891	8/23/2004	12.5	M	0.311
Island	8892	8/23/2004	17.1	F	0.298
Island	8894	8/23/2004	23.8	F	0.518
Lac Vieux Desert	10831	8/11/2004	23.9	F	0.347
Lac Vieux Desert	10833/ 10832**	8/11/2004	19.3	F	0.313
Lac Vieux Desert	10834	8/11/2004	26	F	0.468
Lac Vieux Desert	10835	8/11/2004	13.6	M	0.146
Lac Vieux Desert	10836	8/11/2004	13.1	M	0.107
Lac Vieux Desert	10838	8/11/2004	20.2	F	0.165
Lac Vieux Desert	10839	8/11/2004	16.0	M	0.157
Lac Vieux Desert	10840	8/11/2004	15.6	M	0.158
Lac Vieux Desert	10841	8/11/2004	21.5	F	0.311
Lac Vieux Desert	10842	8/11/2004	12.4	M	0.093
Lac Vieux Desert	10844	8/11/2004	22.0	F	0.155
Lac Vieux Desert	10845	8/11/2004	16.4	M	0.101
Laura	11246	8/18/2004	17.1	M	0.340
Laura	11247	8/18/2004	20.8	F	0.290
Laura	11248	8/18/2004	17.1	F	0.257
Laura	11249	8/18/2004	15.8	M	0.342
Laura	11251	8/18/2004	26.1	M	0.682
Laura	11253	8/18/2004	13.8	M	0.308
Laura	11254	8/18/2004	21.9	F	0.465
Laura	11255	8/18/2004	20.0	F	0.364

Laura	11256	8/18/2004	24.3	F	0.684
Laura	11257	8/18/2004	22.2	M	0.614
Laura	11258	8/18/2004	14.4	M	0.309
Laura	11259	8/26/2004	14.2	M	0.370
Little Star	32	8/31/2004	17.1	M	0.420
Little Star	33	8/31/2004	12.4	M	0.342
Little Star	34	8/31/2004	13.1	M	0.257
Little Star	35	8/31/2004	20.0	F	0.783
Little Star	36	8/31/2004	15.8	F	0.409
Little Star	37	8/31/2004	15.7	M	0.277
Little Star	38	8/31/2004	21.6	F	0.764
Little Star	39	8/31/2004	19.1	F	0.430
Little Star	41	8/31/2004	20.4	F	0.687
Little Star	42	8/31/2004	18.0	F	0.445
Little Star	43	8/31/2004	14.5	M	0.255
Little Star	44	8/31/2004	17.6	F	0.379
Long	10231	8/31/2004	13.2	M	0.254
Long	10232	8/31/2004	19.4	M	0.385
Long	10233	8/31/2004	18.0	M	0.493
Long	10234	8/31/2004	19.2	M	0.397
Long	10235	8/31/2004	14.9	M	0.222
Long	10236	8/31/2004	15.7	M	0.212
Long	10238	8/31/2004	12.8	M	0.329
Long	10240	8/31/2004	17.9	M	0.378
Long	10241	8/31/2004	18.0	M	0.435
Long	10243	8/31/2004	13.9	M	0.227
Long	10244	8/31/2004	18.1	M	0.494
Long	10245	8/31/2004	20.7*	M	0.371
Lucerne	8791	8/4/2004	13.8	M	0.158

Lucerne	8792	8/4/2004	18.2	M	0.342
Lucerne	8795	8/4/2004	19.2	M	0.656
Lucerne	8796	8/4/2004	13.5	M	0.229
Lucerne	8797	8/4/2004	12.9	M	0.216
Lucerne	8798	8/4/2004	20.9	F	0.627
Lucerne	8799	8/4/2004	16.6	M	0.288
Lucerne	8800	8/4/2004	17.7	M	0.391***
Lucerne	8896	8/4/2004	18.1	M	0.515
Lucerne	8898	8/4/2004	21.8	F	1.09
Lucerne	8899	8/4/2004	25.9	F	0.512
Lucerne	8900	8/4/2004	15.8	M	0.268
Manitowish	16	9/17/2004	19.3	F	0.548
Manitowish	18	9/17/2004	17.3	F	0.656
Manitowish	19	9/17/2004	14.9	M	0.245
Manitowish	20	9/17/2004	17.4	F	0.585
Manitowish	21	9/17/2004	13.7	M	0.167
Manitowish	22	9/17/2004	13.0	M	0.157
Manitowish	23	9/17/2004	14.1	M	0.275
Manitowish	25	9/17/2004	13.2	M	0.328
Manitowish	26	9/17/2004	15.6	M	0.164
Manitowish	28	9/17/2004	19.4	F	0.438
Manitowish	29	9/17/2004	16.9	F	0.240
Manitowish	30	9/17/2004	19.5	F	0.391
Middle Eau Claire	10732	8/31/2004	18.3	M	0.456
Middle Eau Claire	10733	8/31/2004	12.3	M	0.176
Middle Eau Claire	10734	8/31/2004	14.6	M	0.271
Middle Eau Claire	10736	8/31/2004	16.3	M	0.367
Middle Eau Claire	10737	8/31/2004	22.8	F	1.02
Middle Eau Claire	10738	8/31/2004	16.4	F	0.373

Middle Eau Claire	10739	8/31/2004	15.0	M	0.484
Middle Eau Claire	10740	8/31/2004	19.1	M	0.689
Middle Eau Claire	10741	8/31/2004	20.0	F	0.652
Middle Eau Claire	10742	8/31/2004	14.6	M	0.282
Middle Eau Claire	10743	8/31/2004	22.1	F	0.377
Middle Eau Claire	10745	8/31/2004	25.5	F	1.13
Nebagamon	10716	8/4/2004	14.1	M	0.452
Nebagamon	10717	8/4/2004	17.9	F	0.691
Nebagamon	10718	8/4/2004	22.1	F	0.693
Nebagamon	10720	8/4/2004	17.8	F	0.619
Nebagamon	10721	8/4/2004	13.6	M	0.516
Nebagamon	10722	8/4/2004	13.2	M	0.729
Nebagamon	10723	8/4/2004	24.0	F	0.887
Nebagamon	10724	8/4/2004	16.7	F	0.460
Nebagamon	10726	8/4/2004	21.0	F	1.18
Nebagamon	10727	8/4/2004	20.1	F	0.754
Nebagamon	10728	8/4/2004	18.9	M	1.03
Nebagamon	10730	8/4/2004	22.5	F	0.953
Nelson	10791	8/23/2004	14.9	M	0.209
Nelson	10797	8/23/2004	18.5	M	0.351
Nelson	10798	8/23/2004	23.1	F	0.496
Nelson	10799	8/23/2004	17.7	M	0.494
Nelson	10800	8/23/2004	19.7	M	0.521
Nelson	11297	8/23/2004	16.6	M	0.415
Nelson	11298	8/23/2004	16.2	M	0.320
Nelson	11299	8/23/2004	18.1	M	0.383
Nokomis	11216	8/17/2004	25.0	F	0.458
Nokomis	11221	8/17/2004	21.5	F	0.837
Nokomis	11222	8/17/2004	14.4	M	0.283

Nokomis	11223	8/17/2004	14.8	M	0.283
Nokomis	11224	8/17/2004	15.5	M	0.307
Nokomis	11225	8/17/2004	17.5	M	0.423
Nokomis	11226	8/17/2004	20.5	F	0.402
Nokomis	11227	8/17/2004	13.2	M	0.176
Nokomis	11228	8/17/2004	18.2	F	0.482
Nokomis	11229	8/17/2004	15.1	M	0.382
Pelican	1831	8/17/2004	23.0*	F	0.374
Pelican	11276	8/17/2004	19.9	F	0.261
Pelican	11279	8/17/2004	15.0	M	0.197
Pelican	11281	8/17/2004	18.6	M	0.260
Pelican	11282	8/17/2004	19.1	M	0.242
Pelican	11283	8/17/2004	13.2	M	0.139
Pelican	11285	8/17/2004	14.3	M	0.116
Pelican	11286	8/17/2004	22.0	F	0.341
Pelican	11287	8/17/2004	16.3	M	0.255
Pelican	11288	8/17/2004	14.9	M	0.130
Pelican	11289	8/17/2004	22.3	F	0.292
Pelican	11290	8/17/2004	17.4	M	0.205
Rest	46	8/17/2004	12.6	M	0.164
Rest	48	8/17/2004	13.1	M	0.136
Rest	49	8/17/2004	17.5	F	0.311
Rest	52	8/17/2004	12.8	M	0.182
Rest	53	8/17/2004	15.5	M	0.158
Rest	59	8/17/2004	17.4	M	0.251
Rest	1840	8/17/2004	21.1	M	0.520
Rest	1843	8/17/2004	18.5	F	0.308
Roberts	10816	8/17/2004	20.7	F	0.396
Roberts	10817	8/17/2004	19.8	F	0.361

Roberts	10818	8/17/2004	16.0	M	0.393
Roberts	10819	8/17/2004	18.3	F	0.346
Roberts	10820	8/17/2004	14.8	M	0.379
Roberts	10822	8/17/2004	15.5	M	0.489
Roberts	10825	8/17/2004	13.5	M	0.249
Roberts	10827	8/17/2004	15.0	M	0.506
Roberts	10828	8/17/2004	12.7	M	0.218
Roberts	10830	8/17/2004	23.6	F	0.617
Round	10861	8/23/2004	15.2	M	0.152
Round	10862	8/23/2004	16.6	M	0.163
Round	10863	8/23/2004	22.3	F	0.290
Round	10864	8/23/2004	13.9	M	0.110
Round	10865	8/23/2004	23.0	F	0.292
Round	10868	8/23/2004	13.0	M	0.089
Round	10869	8/23/2004	18.5	M	0.470
Round	10870	8/23/2004	14.7	M	0.110
Round	10873	8/23/2004	16.0	M	0.240
Round	10874	8/23/2004	19.0	M	0.248
Round	10875	8/23/2004	19.4	M	0.251
Sevenmile	10801	8/26/2004	15.4	M	0.769
Sevenmile	10802	8/26/2004	22.0	F	0.987
Sevenmile	10803	8/26/2004	17.7	M	0.948
Sevenmile	10804	8/26/2004	14.5	M	0.626
Sevenmile	10805	8/26/2004	18.0	M	0.783
Sevenmile	10806	8/26/2004	17.2	M	0.512
Sevenmile	10808	8/26/2004	20.5	F	0.721
Sevenmile	10809	8/26/2004	23.9	F	0.964
Sevenmile	10811	8/26/2004	14.8	M	0.781
Sevenmile	10812	8/26/2004	22.4	F	1.05

Sevenmile	10814	8/26/2004	16.1	M	0.482
Sevenmile	10815	8/26/2004	13.8	M	0.574
Sevenmile	10821	8/26/2004	19.4	F	0.776
Sherman	10216	8/26/2004	12.6	M	0.221
Sherman	10217	8/26/2004	18.0	F	0.440
Sherman	10218	8/26/2004	16.8	M	0.257
Sherman	10219	8/26/2004	23.7	F	0.661
Sherman	10220	8/26/2004	23.7	F	0.733
Sherman	10222	8/26/2004	13.9	M	0.249
Sherman	10223	8/26/2004	12.8	M	0.242
Sherman	10226	8/26/2004	15.4	M	0.220
Sherman	10227	8/26/2004	19.5	M	0.432
Sherman	10228	8/26/2004	24.4	F	0.740
Sherman	10229	8/26/2004	19.5	F	0.423
Sherman	10230	8/26/2004	15.8	M	0.284
Spider (Sawyer)	10876	8/26/2004	18.3	M	0.710
Spider (Sawyer)	10877	8/26/2004	12.0	M	0.289
Spider (Sawyer)	10879	8/26/2004	19.2	M	1.07
Spider (Sawyer)	10880	8/26/2004	14.2	M	0.311
Spider (Sawyer)	10881	8/26/2004	22.2	M	0.587
Spider (Sawyer)	10883	8/26/2004	17.4	M	0.664
Spider (Sawyer)	10884	8/26/2004	14.9	M	0.291
Spider (Sawyer)	10885	8/26/2004	15.8	M	0.254
Spider (Sawyer)	10886	8/26/2004	23.1	F	0.633
Spider (Sawyer)	10887	8/26/2004	19.2	F	0.773
Spider (Sawyer)	10888	8/26/2004	23.6	F	1.11
Spider (Sawyer)	10890	8/26/2004	17.3	M	0.495
Spider (Vilas)	80	8/18/2004	12.2	M	0.337
Spider (Vilas)	87	8/18/2004	14.6	F	0.458

Spider (Vilas)	90	8/18/2004	12.9	M	0.131
Spider (Vilas)	11201	8/18/2004	13.0	M	0.260
Spider (Vilas)	11205	8/18/2004	13.4	M	0.257
Spider (Vilas)	11210	8/18/2004	16.2	F	0.344
Spider (Vilas)	11211	8/18/2004	15.0	M	0.371
Spider (Vilas)	11212	8/18/2004	12.1	M	0.274
Spider (Vilas)	11213	8/18/2004	12.0	M	0.317
Spider (Vilas)	11214	8/18/2004	15.3	M	0.599
Turtle Flambeau Flowage	10891	7/22/2004	15.8	M	0.388
Turtle Flambeau Flowage	10893	7/22/2004	16.1	F	0.660
Turtle Flambeau Flowage	10895	7/22/2004	14.0	M	0.542
Turtle Flambeau Flowage	10897	7/22/2004	16.6	M	0.878
Turtle Flambeau Flowage	10899	7/22/2004	13.7	M	0.565
Turtle Flambeau Flowage	10900	7/22/2004	14.7	M	0.610
Turtle Flambeau Flowage	11294	7/22/2004	21.3	F	1.20
Upper Eau Claire	10746	9/17/2004	16.1	M	0.310
Upper Eau Claire	10748	9/17/2004	27.8	F	1.17
Upper Eau Claire	10749	9/17/2004	18.3	M	0.506
Upper Eau Claire	10750	9/17/2004	19.0	M	0.385
Upper Eau Claire	10751	9/17/2004	14.2	M	0.307
Upper Eau Claire	10752	9/17/2004	16.0	M	0.384
Upper Eau Claire	10754	9/17/2004	27.6	F	1.15
Upper Eau Claire	10755	9/17/2004	13.9	M	0.304
Upper Eau Claire	10756	9/17/2004	14.9	M	0.265

Upper Eau Claire	10758	9/17/2004	19.8	M	0.500
Upper Eau Claire	10759	9/17/2004	17.4	M	0.459
Upper Eau Claire	10760	9/17/2004	22.7	F	0.505
Upper St. Croix	10702	8/26/2004	27.3	F	0.893
Upper St. Croix	10703	8/26/2004	18.5	M	0.601
Upper St. Croix	10705	9/17/2004	17.4	M	0.602
Upper St. Croix	10707	9/17/2004	18.8	M	0.554
Upper St. Croix	10708	9/17/2004	18.3	F	0.690
Upper St. Croix	10709	9/17/2004	17.5	M	0.243
Upper St. Croix	10711	9/17/2004	13.9	M	0.167
Upper St. Croix	10712	9/17/2004	14.9	M	0.206
Upper St. Croix	10713	9/17/2004	17.7	M	0.370
Upper St. Croix	10714	9/17/2004	14.5	M	0.268
Wild Rice	61	8/18/2004	12.5	M	0.242
Wild Rice	62	8/18/2004	12.5	M	0.092
Wild Rice	63	8/18/2004	17.2	F	0.177
Wild Rice	66	8/18/2004	13.9	M	0.448
Wild Rice	67	8/18/2004	14.3	M	0.464
Wild Rice	70	8/18/2004	15.2	F	0.237
Wild Rice	71	8/18/2004	14.6	M	0.510
Wild Rice	72	8/18/2004	14.9	F	0.566
Wild Rice	73	8/18/2004	15.5	M	0.474
Wild Rice	74	8/18/2004	13.3	M	0.254
Wild Rice	75	8/18/2004	13.9	M	0.322

* Frozen length measured by GLIFWC staff.

** Fish 10833 had a label on the sample bag of 10832.

*** Fish 8800 was re-analyzed in duplicate on 1/26/2005. Reported result is the mean of those duplicates.

Table 12. Percent Moisture in Walleye Fillets Measured Immediately After Grinding Coincident with the GLIFWC EPA Fish Analysis.

Lake	Sample ID	Percent Moisture	Duplicate Agreement
Alder	1846	80.7	
Alder	1848	81.6	
Alder	1851	79.8	
Alder	1852	78.3	
Ballard	11263	80.4	
Balsam	11204	78.8	
Balsam	11208	78.6	
Balsam	11209	79.5	
Bass-Patterson	10204	78.8	
Bass-Patterson	10208	80.2	
Bass-Patterson	10211	80.7	
Bass-Patterson	10212	80.8	
Big Portage	11232	78.6	
Big Portage	11232*	79.3	99.1
Big Portage	11233	80.6	
Big Portage	11233*	80.3	99.6
Big Portage	11235	79.2	
Big Portage	11235*	80.1	98.9
Big Portage	11244	78.6	
Big Portage	11244*	79.4	99.0
Big Portage	11244 dup	78.6	100
Big Portage	11244 dup	78.7	99.9
Chippewa	10846	78.5	
Chippewa	10848	78.9	
Chippewa	10850	78.5	

Chippewa	10852	78.0	
Clear	8876	79.6	
Clear	8878	79.0	
Clear	8878 dup	79.3	99.5
Clear	8881	79.9	
Clear	8883	79.1	
Crab	1571	78.9	
Crab	1571*	78.8	99.9
Crab	1572	79.4	
Crab	1575	79.8	
Crab	1575*	80.0	99.8
Crab	1577	79.6	
Crab	1577*	79.0	99.2
Gogebic	10761	80.1	
Gogebic	10763	79.6	
Gogebic	10764	79.7	
Gogebic	10765	80.6	
High	10778	78.4	
High	10778*	78.6	99.7
High	10780	77.8	
High	10780*	78.3	99.4
High	10781	82.5	
High	10781*	82.5	100
High	10782	80.1	
High	10782*	Sample Lost	-
High	10782 dup	79.9	99.7
High	10782 dup*	80.1	100
Island	91	81.6	
Island	93	80.7	

Island	95	80.0	
Island	97	79.4	
Lac Vieux Desert	10831	79.0	
Lac Vieux Desert	10832	79.0	
Lac Vieux Desert	10832 dup	79.0	100
Lac Vieux Desert	10835	80.1	
Lac Vieux Desert	10845	78.9	
Laura	11246	77.8	
Laura	11246 dup	77.4	99.5
Laura	11247	81.4	
Laura	11248	79.1	
Laura	11249	77.5	
Little Star	32	78.9	
Little Star	33	78.2	
Little Star	35	79.8	
Little Star	38	79.7	
Little Star	00038 dup	79.3	99.5
Long	10231	77.3	
Long	10232	78.2	
Long	10233	75.8	
Long	10233 dup	76.0	99.6
Long	10235	78.3	
Lucerne	8796	78.1	
Lucerne	8798	78.2	
Lucerne	8799	78.4	
Lucerne	8799*	78.1	99.6
Lucerne	8800	79.0	
Lucerne	8800*	77.7	98.4
Lucerne	8896	78.3	

Lucerne	8898	79.6	
Manitowish	16	82.5	
Manitowish	19	77.1	
Manitowish	21	79.8	
Manitowish	26	78.9	
Middle Eau Claire	10732	78.9	
Middle Eau Claire	10733	77.0	
Middle Eau Claire	10734	79.0	
Middle Eau Claire	10736	77.5	
Nebagamon	10717	78.7	
Nebagamon	10717*	78.5	99.7
Nebagamon	10717 dup	78.2	99.4
Nebagamon	10718	78.4	
Nebagamon	10718*	78.4	100
Nebagamon	10720	80.3	
Nebagamon	10720*	79.3	98.8
Nebagamon	10730	80.6	
Nebagamon	10730*	80.4	99.8
Nelson	10791	79.1	
Nelson	10799	78.8	
Nelson	11297	79.0	
Nelson	11299	79.0	
Nokomis	11216	79.3	
Nokomis	11221	78.8	
Nokomis	11222	79.5	
Nokomis	11223	78.5	
Pelican	11276	78.8	
Pelican	11279	78.7	
Pelican	11281	78.8	

Pelican	11283	78.8	
Pelican	11283 dup	79.1	99.6
Rest	49	80.6	
Rest	49*	80.8	99.7
Rest	53	78.3	
Rest	53*	78.5	99.7
Rest	59	79.7	
Rest	59*	79.9	99.7
Rest	1840	78.6	
Rest	1840 dup	79.0	99.6
Roberts	10816	80.3	
Roberts	10816 dup	79.9	99.5
Roberts	10818	80.5	
Roberts	10819	79.6	
Roberts	10820	79.4	
Round	10862	79.5	
Round	10864	78.6	
Round	10870	79.3	
Round	10873	79.8	
Sevenmile	10801	79.3	
Sevenmile	10801 dup	79.2	99.9
Sevenmile	10804	79.6	
Sevenmile	10814	79.4	
Sevenmile	10815	79.1	
Sherman	10216	79.4	
Sherman	10216*	79.6	99.7
Sherman	10217	79.8	
Sherman	10217*	80.1	99.6
Sherman	10218	78.1	

Sherman	10218*	78.4	99.6
Sherman	10219	80.6	
Sherman	10219*	80.8	99.7
Spider	10876	78.7	
Spider	10879	78.3	
Spider	10880	79.0	
Spider	10881	79.5	
Spider (Vilas)	11210	79.0	
Spider (Vilas)	11211	80.0	
Spider (Vilas)	11211 dup	79.8	97.5
Spider (Vilas)	11212	81.0	
Spider (Vilas)	11214	80.6	
Turtle Flambeau	10891	83.6	
Turtle Flambeau	10893	79.7	
Turtle Flambeau	10893 dup	79.4	99.5
Turtle Flambeau	10897	75.0	
Turtle Flambeau	10900	80.4	
Upper Eau Claire	10746	78.8	
Upper Eau Claire	10748	79.1	
Upper Eau Claire	10748 dup	79.1	100
Upper Eau Claire	10749	77.6	
Upper Eau Claire	10750	76.9	
Upper St. Croix	1709	79.3	
Upper St. Croix	10703	79.8	
Upper St. Croix	10705	80.3	
Upper St. Croix	10708	80.5	
Wild Rice	61	77.8	
Wild Rice	63	77.4	
Wild Rice	63 dup	77.7	99.6

Wild Rice	66	78.6
Wild Rice	74	81.8
	Grand Mean ± Std. Dev.	79.2 ± 1.19

* The sample was dried 24 hrs, weighed, and returned to drying oven for 24 hrs prior to weighing a second time.

Results of the La Crosse Fish Analysis

Quality Assurance – Mercury analysis of the canned tuna fish from eight occasions coincident with the grinding of walleye collected under this grant resulted in a mean percent agreement of 79.0 ± 12.9 percent agreement (Table 13). All percent agreements were above the minimum acceptance limit of 50 percent.

Analysis of the dogfish shark tissue (DORM-2) standard reference material was conducted with each set of walleye tissues analyzed for a total of ten analyses (Table 14). The certified mercury concentration for the dogfish tissue was 4.64 ± 0.26 µg Hg/g. The grand mean and standard deviation was 93.2 ± 8.32 percent of the certified value. All values were within the acceptance range for DORM-2 samples.

Fish tissues were analyzed in duplicate four times. Two portions of the same tissue were analyzed independently. Agreement between two mercury analyses of the same tissue averaged 93.5 ± 2.99 percent (Table 15). All values were in the acceptance range for percent agreement.

Three samples of tissue were spiked with known concentrations of mercury prior to digestion. Two of the samples (No. 6614 and 9492) had a mean recovery of 95.0 ± 1.78 percent, which is within the quality assurance range of 55 to 115 percent (Table 16). The other spike sample (No. 9653) had mean recoveries of 115.5 and 140.8 percent on subsequent analyses and was determined to have interferences. Therefore, results from this sample will be interpreted with caution.

Mercury Analysis – Skinless filets of 22 fish (twenty walleye and two northern pike) from six lakes were analyzed for total mercury concentration. Total mercury concentrations on a wet weight basis ranged from 0.122 to 0.632 µg Hg/g (parts per million) in walleye and from 0.132 to 0.236 µg Hg/g in northern pike (Table 17).

Tissue Moisture Analysis – Moisture was measured in the muscle of ground filets immediately following grinding (Table 18). Walleye and northern pike muscle tissue contained 79.1 ± 1.17 percent moisture determined in sixteen of the fish samples analyzed (80 %).

Table 13. Percent Agreement of Procedural Blank Samples [Commercial Tuna Fish (Thunnus

sp.)

Samples Before and After Grinding] for Total Mercury Coincident with the La Crosse Fish Analysis.

Date of Analysis	Grinding Date	Before Grinding (µg Hg/g)	After Grinding (µg Hg/g)	Relative Percent Agreement
8/11/2004	7/30/2004	0.075	0.060	77.8
8/11/2004	8/4/2004	0.078	0.074	94.7
8/18/2004	7/20/2004	0.054	0.080	61.2*
8/18/2004	7/27/2004	0.080	0.053	59.4*
8/31/2004	7/27/2004	0.083	0.071	84.4
8/31/2004	7/28/2004	0.089	0.100	88.4
10/13/2004	7/20/2004	0.076	0.061	78.1
10/13/2004	7/27/2004	0.073	0.065	88.4
			Mean ± Std. Dev.	79.0 ± 12.9

* Sample analysis not acceptable; re-analyzed 10/13/04.

Table 14. Mercury Concentrations of Dogfish Tissue Supplied by the National Research Council Canada (DORM-2) Coincident with the La Crosse Fish Tissue Analysis. The Tissue has a Certified Mercury Concentration of 4.64 ± 0.26 µg Hg/g Tissue.

Date of Analysis	Sample #1	Sample #2	Mean	Std. Dev.	Percent of Expected
8/11/2004	4.70	4.09	4.39	0.43	94.7
8/11/2004	4.01	3.97	3.99	0.03	86.1
8/18/2004	5.15	3.88	4.52	0.90	97.3
8/18/2004	4.08	4.06	4.07	0.02	87.7

8/31/2004	3.85	3.71	3.78	0.10	81.4
8/31/2004	4.23	4.68	4.46	0.32	96.1
10/13/2004	4.40	4.27	4.34	0.09	93.4
10/13/2004	5.94	4.14	5.04	1.27	108.6
		Mean ± Std. Dev.	4.32 ± 0.39		93.2 ± 8.32

Table 15. Percent Agreement Between Duplicate Analysis for Total Mercury (Wet Weight) Content in Skinless Filet Tissue of Walleye Muscle Coincident with La Crosse Fish Analysis.

Date of Analysis	Sample ID	Sample #1 (µg Hg/g)	Sample #2 (µg Hg/g)	Mean	Relative Percent Agreement
8/11/2004	Squaw 6614	0.301	0.332	0.316	90.2
8/31/2004	Mille Lacs 9492	0.160	0.155	0.158	96.8
8/26/2004	Squaw 9653	0.532	0.490	0.511	91.8
10/13/2004	Squaw 9653	0.493	0.469	0.481	95.0
				Mean ± Std. Dev.	93.5 ± 2.99

Table 16. Percent of Mercury Recovered from Skinless Walleye Fillet Samples Spiked with a Known Quantity of Mercury Coincident with the Analysis of La Crosse Fish Tissue.

Date of Analysis	Sample ID	Spike #1	Spike #2	Mean	Std. Dev.
8/11/2004	Squaw 6614	96.2	96.1	96.2	0.11
8/31/2004	Mille Lacs	91.2	96.1	93.7	3.45

	9492				
8/26/2004	Squaw 9653	111.3	119.8	115.5*	6.05
10/13/2004	Squaw 9653	136.9	144.7	140.8*	5.52
			Mean ± Std. Dev	112 ± 21.8	

* Sample spike recovery outside of acceptable range, re-analyzed on 10/13/04. Sample still outside of acceptable range suggesting interference in sample.

Table 17. Total Mercury Concentration (Wet and Dry Weight) in Walleye and Northern Pike Fillets from Various Length and Sex Fish for the La Crosse Fish Analysis.

Lake	Sample ID	UW-LaCrosse ID	Species	Date Analyzed	Total Length (in)	Sex	µg Hg/g (Wet Wt.)	µg Hg/g (Dry Wt.)
Bearskin	9633	ERP-BS04WI012	Walleye	8/11/2004	16.7	M	0.122	0.584
Bearskin	9629	ERP-BS04WI006	Walleye	8/11/2004	18.6	M	0.180	0.842
Franklin	9620	ERP-FR04WE001	Walleye	8/18/2004	25.7	F	0.365	1.75
Franklin	9601	ERP-FR04WE004	Walleye	8/18/2004	14.8	M	0.540	2.64
Mille Lacs	9493	ERP-ML04WE009	Walleye	8/31/2004	20.6	F	0.192	0.872
Mille Lacs	9492	ERP-ML04WE007	Walleye	8/31/2004	17.3	M	0.158	0.777
Mille Lacs	1374	ERP-ML04NP009	Northern Pike	8/31/2004	22.6	M	0.132	0.618
Mille Lacs	1381	ERP-ML04NP012	Northern Pike	8/31/2004	39.0	F	0.236	0.976

Squaw	9757	ERP-SQ04WE017	Walleye	10/13/2004	16.1	F	0.541	2.69
Squaw	9653	ERP-SQ04WE018	Walleye	10/13/2004	14.2	M	0.481	2.36
Tomahawk Chain	9684	ERP-TH04WE002	Walleye	8/11/2004	19.9	F	0.216	1.09
Tomahawk Chain	9693	ERP-TH04WE007	Walleye	8/11/2004	24.0	M	0.632	2.86
Squaw 2003	6606		Walleye	8/11/2004	13.8	M	0.465	
Squaw 2003	6608		Walleye	8/11/2004	15.6	M	0.448	
Squaw 2003	6611		Walleye	8/11/2004	13.2	F	0.405	
Squaw 2003	6613		Walleye	8/11/2004	12.9	M	0.452	
Squaw 2003	6614		Walleye	8/11/2004	15.5	F	0.316	
Squaw 2003	6615		Walleye	8/11/2004	14.3	F	0.377	
Squaw 2003	6616		Walleye	8/11/2004	12.7	M	0.309	
Squaw 2003	6617		Walleye	8/11/2004	12.6	M	0.277	
Squaw 2003	6618		Walleye	8/11/2004	15.5	F	0.379	
Squaw 2003	6619		Walleye	8/11/2004	14.7	F	0.450	

Table 18. Percent Moisture in Walleye Fillets Measured Immediately After Grinding Coincident with La Crosse Fish Analysis.

Lake	Sample ID	UW-LaCrosse ID	Percent Moisture	Duplicate Agreement
Bearskin	9633	ERP-BS04WI012	79.1	
Bearskin	9629	ERP-BS04WI006	78.6	
Franklin	9620	ERP-FR04WE001	79.2	
Franklin	9601	ERP-FR04WE004	79.5	
Mille Lacs	9493	ERP-ML04WE009	78.0	
Mille Lacs	9492	ERP-ML04WE007	79.7	
Mille Lacs	1374	ERP-ML04NP009	78.6	
Mille Lacs	1381	ERP-ML04NP012	75.8	
Squaw	9757	ERP-SQ04WE017	79.9	
Squaw	9653	ERP-SQ04WE008	79.6	
Tomahawk Chain	9684	ERP-TH04WE002	80.1	
Tomahawk Chain	9693	ERP-TH04WE007	77.9	
Squaw	6606		80.3	
Squaw	6606 dup		80.5	99.8
Squaw	6608		80.0	
Squaw	6611		79.4	
Squaw	6613		78.1	
		Mean ± Std. Dev.	79.1 ± 1.17	

APPENDIX A

PROCEDURES FOR COLLECTING, PREPARING AND TRANSPORTING FISH SAMPLES

INTRODUCTION

This SOP includes general guidelines for the collection of fish samples at the study sites, preparing the specimens as samples, wrapping and labeling samples, preservation, and transportation to the laboratory for further studies. Species of fish collected may vary, and the preparation of each species may vary slightly, depending on the needs for the analysis to be performed. The objective of this SOP is to provide to the analytical laboratory samples of fish tissue that is properly identified, labeled, wrapped, preserved, and comparable from one sample to the next.

EQUIPMENT LIST

- Permanent Ink Marker
- Solvent Rinsed Aluminum Foil
- Gallon-Size Freezer Bags
- Knives Sufficient to Filet Fish
- Freezer Space for Storage of Samples
- Coolers for Shipment
- Ice for Coolers
- Log Sheet to Record Data
- Label Tape
- Pencil

PROCEDURE

1. Collect fish samples in a manner appropriate for the study.
2. Identify the species of fish for sampling.
3. Prepare a waterproof label to identify each sample (use pencils or indelible ink only).
   a. Label the species.
   b. Label the date of capture.
   c. Label the place (lake) of capture.
   d. Total length and weight of whole fish.
   e. Sex of fish (when necessary or possible).
   f. Other data as required.
4. Prepare the fish as a sample (i.e., whole animal, entrails removed, filet with skin or without skin, etc.).
5. Place sample in acetone- or hexane-rinsed aluminum foil if the sample is to be analyzed for organic materials. Place sample in a plastic bag if the sample is to be analyzed for metals.
6. Dual labels are recommended. Place a waterproof label in the package with the sample and another label on the outside of the package.
7. Place the sample on ice in the field as soon as possible (within two hours) and deliver to a freezer within the same 24-hour period.
8. Record on a separate log (sheet of paper or log book) the data that was included on the labels with the fish samples.
9. Transport sample to the laboratory in frozen condition (do not let samples thaw until ready for analysis).

Example of Label

Name of Study:	Date:
Species:	Location of Capture:
Total Length (units):	Weight (units):
Sex:	Name of Investigator:
Other Information:	

APPENDIX B

STANDARD OPERATING PROCEDURE

COLD VAPOR MERCURY ANALYSIS - ROUTINE LABWARE CLEANING

INTRODUCTION

This cleaning procedure is used for the routine cleaning of labware being used during any cold vapor mercury analysis procedures. The proper safety equipment must be worn during the entire cleaning procedure. This includes gloves, goggles, and lab coat.

EQUIPMENT LIST

- Deionized Water
- Gloves
- Lab Coat
- Micro or Liquinox Detergent
- Various Labware Washing Brushes
- Plastic Dish Rack
- Plastic 14"x10"x10" HPDE tank with cover
- Ammonium Hydroxide, 30% (reagent grade)
- Nitric Acid, Concentrated (Reagent grade)
- Dish Pan
- Goggles
- Labware to be Washed
- pH Indicator Strips
- Wash Bottle

PROCEDURE: LABWARE CLEANING

1. Scrub the labware thoroughly in hot water containing Micro or Liquinox detergent.
2. Rinse the labware with hot water until there is no presence of soap.
3. Rinse the labware once with deionized water.
4. Place the labware in the plastic tank containing 10% nitric acid. Be sure the labware is completely filled with acid. Allow the labware to soak for a minimum of 60 minutes.
5. Remove the labware from the tank, emptying the acid back into the tank.
6. Rinse the labware three times with deionized water.
7. Place the clean labware in a plastic rack to air dry. When the labware is dry, cover the labware with a lid, stopper, or aluminum foil. Place the labware in a proper storage location until used.

PROCEDURE: PLASTIC TANK CONTAINING 10% (V/V) NITRIC ACID

1. Fill the tank with 14.4 liters of deionized water. Then add 1.6 liters of concentrated nitric acid and stir. The tank is now ready to be used to soak labware.
2. Every few months change the acid in the tank. Neutralize the acid with ammonium hydroxide until a pH of between 6 and 10 is achieved. Measure the pH in the tank with pH indicator strips.
3. Pour the neutralized acid down the drain with running cold water. Run the cold water for an additional 10 minutes.
4. Rinse the tank with warm tap water and then with deionized water. Fill the tank with 10% nitric acid as in step 1.

APPENDIX C

STANDARD OPERATING PROCEDURE

COLD VAPOR MERCURY ANALYSIS - MEAT GRINDER CLEANING

INTRODUCTION

This cleaning procedure is only required for meat grinder and labware being used for grinding of fish samples for cold vapor mercury analysis. The proper safety equipment must be worn during the entire cleaning procedure. This includes gloves, goggles, and lab coat.

EQUIPMENT LIST

- Plastic Pan
- Dish Pan
- Goggles
- Liquinox Detergent
- Various Labware Washing Brushes
- Meat Grinder
- Ammonium Hydroxide, 30% (Reagent grade)
- Hydrochloric Acid, Concentrated (Reagent grade)
- Deionized Water
- Gloves
- Lab Coat
- pH Indicator Strips
- Wash Bottle
- Labware to be Washed

PROCEDURE: MEAT GRINDER AND LABWARE CLEANING

1. Dismantle the meat grinder before washing.
2. Scrub the meat grinder components and labware thoroughly in hot water containing Liquinox detergent.
3. Rinse the meat grinder components and labware with hot water until there is no presence of soap.
4. Rinse the meat grinder components and labware with deionized water.
5. Place the meat grinder components and labware in a plastic pan containing 0.1 M HCl. Be sure that the meat grinder components and labware are completely immersed in the acid. Allow the meat grinder components and labware to soak for 30 seconds.
6. Rinse the meat grinder components and labware with deionized water.
7. Assemble the meat grinder which is ready to be used.

PROCEDURE: PLASTIC PAN CONTAINING 0.1 M HYDROCHLORIC ACID

1. Fill the plastic pan with 4 liters of deionized water. Then add 33 mL of concentrated hydrochloric acid and stir. The pan is now ready to be used to soak.
2. Periodically change the acid in the plastic pan. Neutralize the acid with ammonium hydroxide until a pH of between 6 and 10 is achieved. Measure the pH in the plastic pan with pH indicator sticks.
3. Pour the neutralized waste down the drain with running cold water. Run the cold water for an additional five minutes.
4. Rinse the plastic pan with warm tap water and then with deionized water. Fill the plastic pan with 0.1 M hydrochloric acid as in step 1.

APPENDIX D

STANDARD OPERATING PROCEDURE

COLD VAPOR MERCURY ANALYSIS - FISH GRINDING

INTRODUCTION

This procedure is for the grinding of fish filets into homogeneous samples. The meat grinder and labware used to grind the fish is cleaned by the "Cold Vapor Mercury Analysis - Meat Grinder Cleaning (SA/9)" procedure. The jars the ground fish samples are placed in are cleaned by the "Cold Vapor Mercury Analysis - New Labware Cleaning (SA/15)" procedure. The proper safety equipment must be worn during the entire grinding procedure. This includes gloves, goggles, and lab coat.

EQUIPMENT LIST

- Fish Filets Samples
- Gloves
- Lab Coat
- Spatula
- Aluminum Foil
- Tuna fish
- Food Processor with Grinding Attachments
- Filet Knife
- Goggles
- Grinder
- Beaker
- Scintillation Vials

PROCEDURE: GRINDING FISH Filet SAMPLES

1. Cut the fish filets into small pieces that will fit through the grinder feed tube or food processor with grinding attachments.
2. Pass the fish through the grinder or food processor, discarding the first few grams of tissue that come through. Collect the fish tissue in a beaker.
3. Mix the fish tissue with a spatula.
4. Repeat steps 2 and 3 an additional two times.
5. Place the fish in a previously acid-cleaned container. Seal securely with the screw top lid. Label the vial with the appropriate information and place in a freezer until analyzed.
6. Wash the grinder (or food processor) and labware by the "Cold Vapor Mercury Analysis - Meat Grinder Cleaning " procedure before grinding the next fish sample.
7. Continue to grind each fish sample by steps 1 - 7.

PROCEDURE: PREPARING THE PROCEDURAL BLANK

1. Drain a can of tuna fish to be used as the procedural blank. Grind half the tuna fish as a procedural blank by use of steps 2 - 7. Label the tuna fish as "ground" and include with the analysis set.
2. The other half of the tuna is left unground and handled like a sample by use of steps 5 + 6. Label the tuna fish as "unground" and include with the analysis set.

APPENDIX E

COLD VAPOR MERCURY ANALYSIS - FISH SAMPLE WEIGHING

INTRODUCTION

This procedure is for the weighing of ground fish tissue for cold vapor mercury analysis. The fish should be ground by use of the "Cold Vapor Mercury Analysis - Fish Grinding" procedure. The labware used in this procedure should be cleaned by the "Cold Vapor Mercury Analysis - Routine Labware Cleaning" procedure. The proper safety equipment must be worn during this entire procedure. This includes gloves, safety glasses or goggles, and lab coat.

EQUIPMENT LIST

- Ground Fish Samples
- Goggles or Safety Glasses
- Nitric Acid (10%)
- Glass Bottles with Ground Glass Stoppers
- Balance Capable of Reading to the Nearest 0.001 g
- Gloves
- Lab Coat
- Spatula
- Kimwipes

PROCEDURE

1. Remove the fish to be analyzed from the freezer and allow to partially thaw.
2. Check the level of the balance and adjust if necessary. Clean the top of the balance of any foreign materials with a soft brush.
3. Zero the balance with the zero adjustment to read 0.000 g.
4. Place a clean glass bottle on the balance and measure weight. Tare the balance.
5. Weigh approximately 0.2 g - 0.3 g of fish tissue into the glass bottle.
6. Weigh and record the total weight of the glass bottle and fish tissue.
7. Rinse the spatula with water, 10% nitric acid and deionized water. Wipe the spatula clean with a Kimwipe.
8. Label and record each glass bottle and fish sample. Be sure that none of the fish tissue adheres to the side of the glass bottle.

APPENDIX F

FIMS MERCURY ANALYSIS - STOCK, STANDARD AND SPIKE PREPARATION

INTRODUCTION

This procedure is used for the preparation of the stock, analytical standards, blanks and spikes for analysis using the Perkin Elmer FIMS-100 Mercury Analyzer. The fish/tissue used for the spikes should be weighed by the use of the "Sample Weighing for Metals Analysis (SA/11)" procedure. The labware used in this procedure should be cleaned by the "Routine Labware Cleaning for Metals Analysis" (SA/8) procedure.

EQUIPMENT LIST

- Ground Tissue Samples for Spikes
- Class A Pipettes (1 mL and 3 mL)
- Deionized Water
- Pipette Bulb
- 1000 mg/L Mercuric Nitrate Stock/Reference Solution
- Concentrated Hydrochloric Acid (Trace Metal Grade)
- 5% (w/v) Potassium Permanganate ($KMnO_4$)
- Micropipettes and Tips
- Teflon Beakers for Making Substocks
- Mercury Waste Container
- 2 Volumetric Flasks (100 mL)
- Polypropylene Digestion Cups (Environmental Express)

PROCEDURE

1. Pipet 1 mL of a 1000 mg/L mercuric nitrate stock solution into a 100 mL volumetric flask containing ~60 mL of deionized water, 1 mL trace metal grade concentrated HCl, and 100 µL 5% $KMnO_4$. Dilute to 100 mL with deionized water to prepare a 10 mg/L Hg substock. Label this solution with the concentration, date and initials as it must be remade once a month.
2. Pipet 1 mL of the 10 mg/L Hg substock solution into a 100 mL volumetric flask containing ~60 mL of deionized water, 0.5 mL trace metal grade concentrated HCl, and 100 µL 5% $KMnO_4$. Dilute to 100 mL with deionzed water to prepare a 100 µg/L Hg substock. Label this solution with the concentration, date and initials as it must be remade once a week.
3. Pipet the following volumes of deionized water and 100 µg/L Hg substock into digestion cups labeled with the appropriate concentrations which are based on the final volume (50 mL) of standard at time of analysis. Use a micropipette to deliver all water volumes and stock Hg volumes less than 1 mL. Use a class A pipet to deliver 3 mL 100 µg Hg/L substock.

Concentration (ng/L)	Amount of 100 µg/L substock	Amount of DI water
Blank	0	3 mL
50	25 µL	2975 µL
100	50 µL	2950 µL
500	250 µL	2750 µL

1000	500 µL	2500 µL
6000	3 mL	0 mL

4. Each blank and standard should be prepared in duplicate.
5. A total of 10% of samples analyzed for mercury should be spiked in duplicate. Spiking is accomplished by pipetting a known volume of the 100 µg/L Hg substock into a digestion cup containing a known weight of fish tissue. A micropipette may be used to deliver two 750 µL aliquots onto pre-weighed tissue to give a total spiking volume of 1.5 mL.
6. All mercury waste from rinsing pipettes, beakers, etc. should be disposed of in mercury waste container. Volume and concentration placed in waste container should be recorded on the hazardous waste container inventory form for that bottle.

APPENDIX G

COLD VAPOR MERCURY DETERMINATION IN BIOTA

INTRODUCTION

This procedure is used for the determination of total mercury in fish, hair and other tissue samples. Do not use this procedure for analyzing human blood.

REFERENCES

"Determination of Mercury in Tissues by Cold Vapor Atomic Absorption Spectrometry", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, April 1991.

EQUIPMENT LIST

- Stannous Chloride, Analytical Reagent
- Magnesium Perchlorate, Anhydrous for Elemental Analysis
- Potassium Persulfate, Reagent Suitable for Mercury Determination
- Hydroxylamine Hydrochloride, Reagent Suitable for Mercury Determination
- Potassium Permanganate, Certified A.C.S.
- Sodium Chloride, Certified A.C.S.
- Sulfuric Acid, A.C.S. Reagent, Suitable for Mercury Determination
- Hydrochloric Acid, Trace Metals Grade
- Nitric Acid, Fisher, Trace Metals Grade
- Mercury Cold Vapor Analyzer
- Hollow Cathode Mercury Lamp
- Variable Autotransformer
- Neptune Dyna-Pump Model 4K
- Hot Block (Environmental Express)
- Varian SpectrAA 200 Spectrophotometer
- FIMS-100 (Perkin Elmer) Mercury Analyzer
- Labindustries Repipet II Dispenser, 3 - 10 mL and 1 - 5 mL
- Wheaton Instruments Socorex Dispenser Model 511, 10 mL
- Polypropylene Digestion Cups and Covers
- Pipets/Pipettors
- Beakers
- Spatulas
- 5% (w/v) Potassium Permanganate
- 5% (w/v) Potassium Persulfate
- 10% (w/v) Hydroxylamine Hydrochloride-10%(w/v) Sodium Chloride
- 10% (w/v) Stannous Chloride-0.5M Sulfuric Acid for Spectra AA Analysis
- 0.05M Potassium Permanganate-5% (v/v) Sulfuric Acid
- 1000 ug/mL Mercuric Nitrate Stock
- 5 ug/mL Mercuric Nitrate Substock for Spectra AA Analysis
- 50 ng/mL Mercuric Nitrate Substock for Spectra AA Analysis
- 10 mg/L Mercuric Nitrate Substock for FIMS-100 Analysis
- 100 ug/L Mercuric Nitrate Substock for FIMS-100 Analysis
- Silicon Defoaming Agent (Perkin Elmer)
- Deionized Water in Teflon Squirt Bottle

PROCEDURE

Digestion

1. Add 4.0 mL of concentrated sulfuric acid and 1.0 mL of concentrated nitric acid to each sample, standard, spike, duplicate and blank.
2. Place the digestion cups in Hot Block at 110°C and allow to digest for approximately 15 minutes or until all the fish tissue is dissolved.
3. Turn off the Hot Block and allow the digestion cups to cool to room temperature.
4. Add 5.0 mL of 5% potassium permanganate to each bottle in 1.0 mL increments swirling the digestion cups after each addition.
5. Add 10.0 mL of 5% potassium permanganate to each digestion cup in 5.0 mL increments, swirling the digestion cup after each addition. Additional 5% potassium permanganate solution (maximum of 5 mL) or solid potassium permanganate should be added to the samples if necessary so that the samples remain purple in color for at least 15 minutes. If extra potassium permanganate is added to a sample, an equal amount should be added to one set of standards and a blank.
6. Add 8 mL of 5% potassium persulfate to each digestion cup, and cover and swirl.
7. Allow the digestion cup to set overnight to oxidize organic mercury compounds to inorganic mercury ions.
8. The samples will remain stable for several days before analysis.

Sample Analysis Using Varian SpectraAA 200

Instrument Conditions

Current = 3.0 mA
Atomic Absorption Mode (AA)
Statistics = 99
D_2 Background Correction with diffraction grating filter
Circulating Pump autotransformer = 70% power
Wavelength = 253.7 nm
Double Beam Mode (DB)
Integration = 1.0 seconds

Instrument Conditions for Varian SpectrAA 200

Sampling Mode = AutoMix
Calibration Mode = Scale Expansion
Measurement Mode = Integrate
Replicates Standard = 20
Replicates Sample = 20
Expansion Factor 1.0
Minimum Reading = Disabled
Smoothing = 9 pt
Conc. Units = ng
Conc. Decimal places = 2
Wavelength = 253.7 nm
Slit Width = 1.0 nm
Lamp Current = 3.0 mA
Background Correction = BC on
Cal. Zero Rate = 0
Measurement Time = 4.5 s
Pre-Read Delay = 0 s
Vapor Type = Cold Vapor
Burner Height = 16.0 mm

1. Set the AA to the instrument conditions listed above and allow instrument warm-up time. Prepare the 10% stannous chloride/0.5 M sulfuric acid solution and the magnesium perchlorate drying tube. Attach the drying tube in the cold vapor mercury analyzer.
2. Autozero the AA by aerating deionized water through the cold vapor mercury analyzer.
3. Transfer the sample from the digestion cup to a glass bottle. Add 10 mL of hydroxylamine hydrochloride/10% sodium chloride to the digestion cup, then transfer to the glass bottle with the sample. Swirl sample until no purple or brown color remains. Rinse the digestion cup with three portions of deionized water, adding the rinse to the sample in the glass bottle each time. Be careful not to end up with the bottle more than two-thirds full.

4. Add 5.0 mL of 10% stannous chloride/0.5 M sulfuric acid to a sample and immediately attach to the mercury analyzer.
5. Measure the absorbance of the sample until the maximum absorbance is reached and begins to decline and record the maximum absorbance as the response.
6. Change the valves of the mercury analyzer to draw the mercury into a 0.05 M potassium permanganate/5% sulfuric acid trap. Purge the mercury analyzer of mercury until the absorbance reaches a minimum similar to the background absorbance.
7. Return the valves to the "analyze" position and rinse the aerator with deionized water before analyzing the next sample. Dispose of the analyzed and purged sample into an Acid Waste container.
8. Alternate analyzing the samples, standards and blanks by use of steps 3-7.
9. Neutralize the "Acid Waste" in a fume hood with ammonium hydroxide until the pH is between 6 and 10. Pour the neutralized waste down the drain with running cold water. Record the volume of waste neutralized in the Acid/Base Waste Log.
10. Collect the exhausted stocks and standards in a glass bottle identified as "Hazardous Waste - Mercuric Nitrate in % acid solutions. Corrosive Toxic." Note the start date. Each waste bottle will require an analysis before it will be accepted for disposal.

Sample Analysis Using Perkin Elmer FIMS-100 Flow Injection Mercury Analysis System

- Prepare the following:
  Carrier Solution (3% HCl)
  Reductant Solution (5% $SnCl_2$, 1% Silicon Defoaming Agent, in 3% HCl)
  Weigh 50g $SnCl_2$ and add to 990 mL 3% HCl. Add 10 mL Silicon Defoaming Agent using 5 mL micropipettor.
- Turn on computer and printer.
- Turn on Nitrogen (400 psi).
- Turn on FIMS 100 mercury analyzer and allow to warm up for 10 minutes minimum.
- Press Ctrl+Alt+Del (on computer).
- Username: administrator.
- Leave password field blank. Click on "OK".
- Open appropriate project Excel file prepared from Hg Calculations-Master and minimize the Excel window.
- Double click on AA Winlab Analyst icon.
- Choose "Use a custom designed workspace".
- Choose "Hg.fms" > "file" > "open" > "method" > "Hg Analysis".
- Click on "Browse" in Results Data Set window and enter a new data set name (DateProject). Be sure that the save data and print log boxes are both checked.
- Turn clamps on the peristaltic pump rollers in order to allow pump to work.
- Check filter compartment cover to see that it has been tightened.
- Attach tubing from filter compartment to cell.
- Click on Manual button (on top toolbar).
- Click on FIAS button (on top toolbar). Run FIAS once using clean deionized water (Click on the "FIAS on/off" button). Place collection tubes into appropriate solution bottles (Red = Reductant solution, Yellow = Carrier Solution) and run FIAS two more times checking the flow of the instrument and the lines for bubbles while it is running. Remember while running a sample set to periodically check carrier and reductant volumes, so they do not deplete.
- Just prior to analysis of all blanks, standards and samples (steps 19-22), add 10 mL of 10% (w/v) Hydroxylamine Hydrochloride - 10% (w/v) Sodium Chloride in two 5 mL aliquots, mix sample until no purple or brown color remains. Dilute to 50 mL with deionized water using the correct line on the digestion cup.
- Rinse the collection tube with deionized water and place in the blank solution. Click on "analyze blank" and allow instrument time to complete triplicate analysis.

- Rinse the collection tube with deionized water and place in the lowest standard. Choose appropriate standard concentration and click on "analyze standard" and allow instrument time to complete triplicate analysis. In the appropriate Excel file for that project, enter 0.000 for the blank absorbance and enter the mean Blank Corrected Signal value for the standard. Repeat this step for each of the five standards to be run in order of lowest to highest to develop the standard curve.
- Rinse the collection tube with deionized water and place in appropriate sample. Enter sample ID code into the appropriate field. Rinse the collection tube with DI water and place in appropriate sample. Click on "analyze sample" and allow instrument time to complete triplicate analysis. Enter the mean Blank Corrected Signal value into the appropriate Excel file for that project. Repeat this step for each of the samples to be analyzed.
- The second Blank, second set of standards, and Dorm-2 samples should be run as they were above, sometime in between samples, to check the precision of the instrument. For example, if the sample set contains 52 samples, including duplicates and spikes, run the first set of standards (~13 samples), the Blank and the lowest standard (50 ng/L), Dorm 2-1 (1) and (2) (~13 samples), the next two standards (100 ng/L and 500 ng/L), Dorm 2-2 (1) (~13 samples), the last two standards (1000 ng/L and 6000 ng/L) and finally Dorm 2-2 (2). It is best to try to analyze the duplicates and spikes without interruption, so more or less than 13 samples may be analyzed between standards in order to keep the samples together and in order.

WHEN ANALYSIS OF ALL SAMPLES AND STANDARDS IS COMPLETE:

- Place sample collection tube, and lines from reductant and carrier solutions into beaker of deionized water.
- Flush/clean tubing with deionized water by running FIAS two times.
- Lift collection tubing out of deionized water and run FIAS one more time to allow air to pass through all tubing. When FIAS is finished running, place collection tubing back into beaker of DI water for storage.
- Raise waste lines out of liquid in waste container so liquid does not back up.
- Release the peristaltic pump rollers so that tubing is not compressed.
- Detach line from cell.
- Unscrew the filter compartment cover and, using forceps to handle filter, dry filter with a Kimwipe.
- Print report. Choose "file" > "utilities" > "reporter" . "Open Design"
  Choose "WR01 Mussel" (double-click), then double-click on the number 1 under result name and choose the data set for that day. Click "OK" > "Print Report" and close the reporter window.
- Save Excel file to floppy disk.
- Turn off FIMS instrument, computer, nitrogen, gas and printer.
- Record the date, project, analyst, number of injections, and time run in FIMS-100 usage record book located on top of instrument.

APPENDIX H

PROCEDURES FOR DETERMINING PERCENT MOISTURE IN TISSUE SAMPLES

INTRODUCTION

This SOP includes general guidelines for the analysis of tissue samples for moisture content. It is a gravimetric technique requiring careful weighing techniques.

EQUIPMENT LIST

- Analytical Balance (i.e., Mettler AG245, PB303, AB204, H34, H72 and H80)
- Aluminum Weighing Pans
- Drying Oven (60° C)
- Desiccation Container
- Spatula

PROCEDURE

1. Calibrate analytical balance using Class One weights. Label the aluminum weighing pans and dry at 60° C for 16 hours.
2. Place dried weighing pans in desiccator until cool.
3. Weigh the dried and cooled weighing pans on an analytical balance to the 0.0001 g.
4. Weigh approximately 1.0 g of thawed tissue and place in the labeled weighing pan.
5. Weigh the pan and the tissue on an analytical balance to the nearest 0.0001 g.
6. Dry pan and tissue in drying oven at 60° C for 16 hours or until constant dry weight is achieved.
7. Remove dried pans and tissue from the oven and place in desiccator until cool.
8. Weigh the pan with the tissue on an analytical balance to the nearest 0.0001 g.
9. It may be necessary to dry the pan and tissue a second time when the tissue is a large mass. Desiccate and reweigh to prove that an equilibrium dry weight has been achieved.
10. Calculations:
    Dry Aluminum Pan - Aluminum pan with wet tissue = Wet weight of tissue
    (Aluminum pan and wet tissue weight - Aluminum pan and dry tissue /
    Wet tissue

APPENDIX I

ABSORBANCES USED FOR DEVELOPMENT OF STANDARD CURVES WITH 2004 LSRI MERCURY ANALYSIS

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0009	0.0014	0.0012	0.0004
100	0.0019	0.0027	0.0023	0.0006
500	0.0098	0.0091	0.0095	0.0005
1000	0.0218	0.0195	0.0207	0.0016
6000	0.1223	ran out of SnCL2 Did not run.	0.1223	-
		corr	0.999969	
		slp	2.04E-05	
		Int	-1.5E-05	

29-Jun-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0011	0.0014	0.0013	0.0002
100	0.0029	0.0029	0.0029	0.0000
500	0.0143	0.0138	0.0141	0.0004
1000	0.0273	0.0274	0.0274	0.0001
6000	0.1557	0.1657	0.1607	0.0071
		corr	0.999988	
		slp	2.68E-05	
		Int	0.000264	

1-Jul-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0012	0.0011	0.0012	0.0001
100	0.0027	0.0025	0.0026	0.0001
500	0.0125	0.0101	0.0113	0.0017
1000	0.0249	0.0232	0.0241	0.0012
6000	0.1436	0.134	0.1388	0.0068
		corr	0.999971	
		slp	2.31E-05	
		Int	0.000172	

7-Jul-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0016	0.0015	0.0016	0.0001
100	0.0031	0.0024	0.0028	0.0005
500	0.015	0.0127	0.0139	0.0016
1000	0.0287	0.0281	0.0284	0.0004

6000	0.1717	0.1621	0.1669	0.0068
		corr	0.999993	
		slp	2.78E-05	
		Int	0.000121	

22-Jul-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0015	0.0016	0.0016	0.0001
100	0.003	0.0029	0.0030	0.0001
500	0.0149	0.0142	0.0146	0.0005
1000	0.0295	0.0288	0.0292	0.0005
6000	0.1721	0.1649	0.1685	0.0051
		corr	0.999981	
		slp	2.8E-05	
		Int	0.000358	

4-Aug-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0015	0.0014	0.0015	0.0001
100	0.0029	0.0028	0.0029	0.0001
500	0.0138	0.0109	0.0124	0.0021
1000	0.0259	0.0212	0.0236	0.0033
6000	0.1597	0.1725	0.1661	0.0091
		corr	0.999666	
		slp	2.78E-05	
		Int	-0.00101	

11-Aug-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0013	0.0012	0.0013	0.0001
100	0.0025	0.0022	0.0024	0.0002
500	0.0133	0.0118	0.0126	0.0011
1000	0.0271	0.0234	0.0253	0.0026
6000	0.1583	0.1396	0.1490	0.0132
		corr	0.999995	
		slp	2.48E-05	
		Int	7.68E-05	

17-Aug-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0012	0.0013	0.0013	0.0001
100	0.0027	0.0029	0.0028	0.0001
500	0.0138	0.0142	0.0140	0.0003
1000	0.0266	0.0269	0.0268	0.0002
6000	0.1526	0.1697	0.1612	0.0121
		corr	0.999992	

slp 2.68E-05
Int 0.000101

18-Aug-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0013	0.0011	0.0012	0.0001
100	0.0022	0.0026	0.0024	0.0003
500	0.0132	0.0112	0.0122	0.0014
1000	0.0256	0.0225	0.0241	0.0022
6000	0.1542	0.1257	0.1400	0.0202

corr 0.999984
slp 2.33E-05
Int 0.000261

23-Aug-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.001	0.0011	0.0011	0.0001
100	0.0024	0.0025	0.0025	0.0001
500	0.0118	0.0116	0.0117	0.0001
1000	0.024	0.0251	0.0246	0.0008
6000	0.1401	0.1375	0.1388	0.0018

corr 0.999945
slp 2.31E-05
Int 0.000288

26-Aug-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0012	0.0012	0.0012	0.0000
100	0.0024	0.0025	0.0025	0.0001
500	0.0114	0.0127	0.0121	0.0009
1000	0.0246	0.0245	0.0246	0.0001
6000	0.1412	0.1367	0.1390	0.0032

corr 0.999951
slp 2.31E-05
Int 0.000384

31-Aug-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0014	0.0013	0.0014	0.0001
100	0.0026	0.0028	0.0027	0.0001
500	0.0133	0.0132	0.0133	0.0001
1000	0.0267	0.0259	0.0263	0.0006
6000	0.1533	0.1431	0.1482	0.0072

corr 0.999940
slp 2.46E-05
Int 0.000546

17-Sep-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0012	0.0011	0.0012	0.0001
100	0.0027	0.0017	0.0022	0.0007
500	0.0136	0.0133	0.0135	0.0002
1000	0.0271	0.0276	0.0274	0.0004
6000	0.1414	0.1398	0.1406	0.0011
		corr	0.999561	
		slp	2.34E-05	
		Int	0.001017	

13-Oct-04

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	0	0.0000	0.0000
50	0.0011	0.0011	0.0011	0.0000
100	0.0023	0.0022	0.0023	0.0001
500	0.0114	0.0119	0.0117	0.0004
1000	0.0226	0.0234	0.0230	0.0006
6000	0.133	0.1445	0.1388	0.0081
		corr	0.999999	
		slp	2.31E-05	
		Int	-3.1E-05	

25-Jan-05

Standard	Abs. 1	Abs. 2	MEAN	STD
0	0	*	0	
50	0.001	*	0.001	
100	0.002	*	0.002	
500	0.0111	*	0.0111	
1000	0.0222	*	0.0222	
6000	0.1295	*	0.1295	
		corr	0.999983	
		slp	2.15E-05	
		Int	0.00012	

* Only one set of standards was run because the set was only 4 samples.

APPENDIX J

2004 RESULTS FOR DETERMINING INSTRUMENT DETECTION (LOD) AND QUANTITATION (LOQ) LIMITS FOR TOTAL MERCURY AT LSRI

Number of Replicates	Degrees of Freedom	t Value	
7	6	3.143	When calculating detection limits a minimum of seven replicates is required. The analyte should not exceed ten times the expected detection limit.
8	7	2.998	
9	8	2.896	
10	9	2.821	
11	10	2.764	
16	10	2.602	
21	20	2.528	
26	25	2.485	t-value x std. Dev. = detection limit (LOD)
31	30	2.457	
61	60	2.39	LOQ = 10/3 x LOD
0	0	2.326	

Analyzed February 10, 2004

Sample	Tissue Type	ng/l	ng Hg	g sample	ug/g			
RF DL-1	rice flour	23.6	1.180	0.500	0.00708			
RF DL-2	rice flour	18.9	0.944	0.501	0.00659			
RF DL-3	rice flour	70.8	3.539	0.514	0.00688			
RF DL-4	rice flour	61.3	3.067	0.498	0.00616			
RF DL-5	rice flour	61.3	3.067	0.502	0.00611			
RF DL-6	rice flour	70.8	3.539	0.505	0.00701	**STDS**	**DL (ug/g)**	**LOQ**
RF DL-7	rice flour	61.3	3.067	0.500	0.00613	0.00042	0.001258	0.004194
RF DL-8	rice flour	70.8	3.539	0.511	0.00693			

Hg LOD =0.00126ug/g
LOQ=0.004194ug/g

Appendix 9

2004 Walleye and Northern Pike Total Mercury Concentrations from the University of Wisconsin-La Crosse Mercury Laboratory

Appendix 9. 2004 Walleye and Northern Pike Total Mercury Concentrations from the University of Wisconsin-La Crosse Mercury Laboratory.

Lake	Tag number	Species	Date Collected	Date of Fish Dissection	Fresh Length (in)	Fish Weight+ Tag (grams)	Sex	Hg Conc Dry Wt (ng/g)	Hg Conc Wet Wt (ng/g)	Hg Conc Wet Wt (ug/g)
Bearskin	09627	Walleye	4/19/2004	6/22/2004	12.5	270	M	458	98	0.098
Bearskin	09629	Walleye	4/21/2004	6/22/2004	18.6	1010	M	874	197	0.197
Bearskin	09630	Walleye	4/19/2004	6/22/2004	13.3	350	M	586	126	0.126
Bearskin	09631	Walleye	4/21/2004	6/22/2004	18.0	830	M	1340	290	0.290
Bearskin	09632	Walleye	4/19/2004	6/22/2004	12.0	230	M	703	151	0.151
Bearskin	09633	Walleye	4/19/2004	6/22/2004	16.7	690	M	600	133	0.133
Bearskin	09634	Walleye	4/21/2004	6/22/2004	18.7	910	M	1187	258	0.258
Bearskin	09635	Walleye	4/19/2004	6/22/2004	13.1	310	M	526	118	0.118
Bearskin	09636	Walleye	4/19/2004	6/22/2004	15.9	530	M	779	172	0.172
Bearskin	09637	Walleye	4/21/2004	6/22/2004	16.0	570	M	595	134	0.134
Bearskin	09638	Walleye	4/19/2004	6/22/2004	19.0	920	M	1201	261	0.261
Bearskin	09639	Walleye	4/19/2004	6/22/2004	15.9	570	M	740	159	0.159
Bearskin	09640	Walleye	4/19/2004	6/22/2004	15.9	580	M	906	193	0.193
Bearskin	09643	Walleye	4/19/2004	6/22/2004	12.9	270	M	320	72	0.072
Bearskin	09644	Walleye	4/21/2004	6/22/2004	18.6	910	M	1050	232	0.232
Bearskin	09645	Walleye	4/21/2004	6/22/2004	19.3	980	M	1195	264	0.264
Bearskin	09647	Walleye	4/21/2004	6/22/2004	16.0	540	M	1372	307	0.307
Bearskin	09648	Walleye	4/19/2004	6/22/2004	19.8	1160	M	1660	375	0.375
Bearskin	09649	Walleye	4/19/2004	6/22/2004	16.9	730	M	752	165	0.165
Franklin	09601	Walleye	4/28/2004	6/17/2004	14.8	500	M	422	90	0.090
Franklin	09602	Walleye	4/28/2004	6/17/2004	18.9	970	F	590	125	0.125
Franklin	09604	Walleye	4/28/2004	6/17/2004	24.1	2670	F	1496	323	0.323
Franklin	09605	Walleye	4/28/2004	6/17/2004	16.9	790	M	502	101	0.101
Franklin	09607	Walleye	4/28/2004	6/17/2004	15.4	560	M	394	83	0.083
Franklin	09613	Walleye	4/28/2004	6/17/2004	19.5	1290	F	731	159	0.159
Franklin	09618	Walleye	4/28/2004	6/17/2004	16.6	640	M	601	121	0.121
Franklin	09619	Walleye	4/28/2004	6/17/2004	13.4	380	M	423	92	0.092
Franklin	09620	Walleye	4/28/2004	6/17/2004	25.7	2580	F	2242	470	0.470
Franklin	09621	Walleye	4/28/2004	6/17/2004	12.4	280	M	321	69	0.069
Franklin	09622	Walleye	4/28/2004	6/17/2004	25.5	3080	F	1707	367	0.367
Franklin	09623	Walleye	4/28/2004	6/17/2004	19.6	1200	F	713	149	0.149
Mille Lacs	09474	Walleye	4/23/2004	6/22/2004	18.6	970	M	639	144	0.144
Mille Lacs	09475	Walleye	4/23/2004	6/22/2004	14.5	420	M	633	128	0.128
Mille Lacs	09477	Walleye	4/23/2004	6/22/2004	18.0	850	M	474	106	0.106
Mille Lacs	09479	Walleye	4/23/2004	6/22/2004	14.2	400	M	439	100	0.100

Lake	Tag number	Species	Date Collected	Date of Fish Dissection	Fresh Length (in)	Fish Weight+ Tag (grams)	Sex	Hg Conc Dry Wt (ng/g)	Hg Conc Wet Wt (ng/g)	Hg Conc Wet Wt (ug/g)
Mille Lacs	09481	Walleye	4/23/2004	6/22/2004	14.9	430	M	399	87	0.087
Mille Lacs	09482	Walleye	4/23/2004	6/22/2004	17.4	710	M	748	164	0.164
Mille Lacs	09483	Walleye	4/23/2004	6/22/2004	17.3	800	M	649	143	0.143
Mille Lacs	09486	Walleye	4/23/2004	6/22/2004	24.5	2440	F	1570	340	0.340
Mille Lacs	09487	Walleye	4/23/2004	6/22/2004	19.0	1110	M	613	133	0.133
Mille Lacs	09488	Walleye	4/23/2004	6/22/2004	14.6	410	M	585	132	0.132
Mille Lacs	09489	Walleye	4/23/2004	6/22/2004	23.7	1780	F	1475	282	0.282
Mille Lacs	09490	Walleye	4/23/2004	6/22/2004	24.1	1430	M	1497	321	0.321
Mille Lacs	09491	Walleye	4/23/2004	6/22/2004	16.5	650	M	585	124	0.124
Mille Lacs	09492	Walleye	4/23/2004	6/22/2004	17.3	740	M	592	129	0.129
Mille Lacs	09493	Walleye	4/23/2004	6/22/2004	20.6	1120	F	772	166	0.166
Mille Lacs	09494	Walleye	4/23/2004	6/22/2004	23.5	1880	F	1381	280	0.280
Mille Lacs	09495	Walleye	4/23/2004	6/22/2004	14.6	440	M	460	100	0.100
Mille Lacs	09496	Walleye	4/23/2004	6/22/2004	22.3	1520	F	1992	435	0.435
Mille Lacs	09497	Walleye	4/23/2004	6/22/2004	22.7	2090	F	1279	277	0.277
Mille Lacs	09498	Walleye	4/23/2004	6/22/2004	17.9	760	M	771	168	0.168
Squaw	09653	Walleye	4/22/2004	6/17/2004	14.2	470	M	3169	629	0.629
Squaw	09654	Walleye	4/22/2004	6/17/2004	15.7	540	M	2351	479	0.479
Squaw	09655	Walleye	4/22/2004	6/17/2004	13.0	320	M	2881	572	0.572
Squaw	09658	Walleye	4/22/2004	6/17/2004	15.6	600	F ?	2258	448	0.448
Squaw	09663	Walleye	4/22/2004	6/17/2004	15.8	600	M	3308	677	0.677
Squaw	09666	Walleye	4/22/2004	6/17/2004	13.5	320	M	3335	625	0.625
Squaw	09673	Walleye	4/23/2004	6/17/2004	17.9	830	M	3071	626	0.626
Squaw	09675	Walleye	4/23/2004	6/17/2004	16.9	720	M	3565	725	0.725
Squaw	09751	Walleye	4/21/2004	6/17/2004	13.3	330	M	2463	480	0.480
Squaw	09752	Walleye	4/21/2004	6/17/2004	19.2	1130	M	2946	618	0.618
Squaw	09754	Walleye	4/21/2004	6/17/2004	15.0	460	F?	2703	515	0.515
Squaw	09755	Walleye	4/21/2004	6/17/2004	12.8	290	M	1056	215	0.215
Squaw	09756	Walleye	4/21/2004	6/17/2004	12.7	270	M	2042	409	0.409
Squaw	09757	Walleye	4/21/2004	6/17/2004	16.1	670	F	3034	563	0.563
Squaw	09759	Walleye	4/21/2004	6/17/2004	13.5	370	M	2258	502	0.502
Squaw	09760	Walleye	4/21/2004	6/17/2004	12.0	230	M	1721	339	0.339
Squaw	09761	Walleye	4/21/2004	6/17/2004	14.1	430	M	1652	341	0.341
Squaw	09767	Walleye	4/21/2004	6/17/2004	15.3	530	F	3758	695	0.695
Squaw	09768	Walleye	4/21/2004	6/17/2004	15.0	460	F	4206	745	0.745
Squaw	09770	Walleye	4/21/2004	6/17/2004	12.3	270	M	1613	322	0.322
Squaw	09772	Walleye	4/21/2004	6/17/2004	12.6	280	M	2049	403	0.403
Squaw	09773	Walleye	4/21/2004	6/17/2004	15.6	630	F?	1772	365	0.365

Lake	Tag number	Species	Date Collected	Date of Fish Dissection	Fresh Length (in)	Fish Weight+ Tag (grams)	Sex	Hg Conc Dry Wt (ng/g)	Hg Conc Wet Wt (ng/g)	Hg Conc Wet Wt (ug/g)
Tomahawk	09676	Walleye	4/29/2004	6/22/2004	21.7	1520	F	1352	349	0.349
Tomahawk	09677	Walleye	4/29/2004	6/22/2004	15.8	570	M	1074	313	0.313
Tomahawk	09678	Walleye	4/29/2004	6/22/2004	18.6	890	F	1091	275	0.275
Tomahawk	09679	Walleye	4/29/2004	6/22/2004	16.3	580	M	1003	301	0.301
Tomahawk	09680	Walleye	4/29/2004	6/22/2004	20.2	1110	F	1267	319	0.319
Tomahawk	09681	Walleye	4/29/2004	6/22/2004	14.8	490	M	1161	347	0.347
Tomahawk	09682	Walleye	4/29/2004	6/22/2004	12.8	310	M	895	353	0.353
Tomahawk	09683	Walleye	4/29/2004	6/22/2004	17.5	790	M	1132	318	0.318
Tomahawk	09684	Walleye	4/29/2004	6/22/2004	19.9	990	F	1038	287	0.287
Tomahawk	09685	Walleye	4/29/2004	6/22/2004	13.8	320	M	616	206	0.206
Tomahawk	09686	Walleye	4/29/2004	6/22/2004	17.7	760	F?	781	216	0.216
Tomahawk	09687	Walleye	4/29/2004	6/22/2004	17.6	660	M	756	218	0.218
Tomahawk	09688	Walleye	4/29/2004	6/22/2004	13.5	310	M	904	316	0.316
Tomahawk	09690	Walleye	4/29/2004	6/22/2004	23.6	1730	F	4058	914	0.914
Tomahawk	09691	Walleye	4/29/2004	6/22/2004	15.3	520	M	1136	350	0.350
Tomahawk	09692	Walleye	4/29/2004	6/22/2004	16.0	580	M	1186	333	0.333
Tomahawk	09693	Walleye	4/29/2004	6/22/2004	24.0	2150	M	3057	794	0.794
Tomahawk	09694	Walleye	4/29/2004	6/22/2004	17.2	710	M	1086	342	0.342
Tomahawk	09695	Walleye	4/29/2004	6/22/2004	19.7	1070	F	1133	297	0.297
Tomahawk	09697	Walleye	4/29/2004	6/22/2004	14.6	410	M	1077	354	0.354
Mille Lacs	1367	Northern Pike	4/23/2004	6/17/2004	30.5	3440	F	594	142	0.142
Mille Lacs	1370	Northern Pike	4/23/2004	6/17/2004	26.1	1870	F	1617	340	0.340
Mille Lacs	1384	Northern Pike	4/23/2004	6/17/2004	24.1	1420	F	727	158	0.158
Mille Lacs	1365	Northern Pike	4/23/2004	6/17/2004	25.2	1470	M	1296	262	0.262
Mille Lacs	1375	Northern Pike	4/23/2004	6/17/2004	23.5	1310	F	338	75	0.075
Mille Lacs	1380	Northern Pike	4/23/2004	6/17/2004	29.4	3120	F	724	155	0.155
Mille Lacs	1382	Northern Pike	4/23/2004	6/17/2004	24.5	1570	M	1077	234	0.234
Mille Lacs	1377	Northern Pike	4/23/2004	6/17/2004	21.1	970	F	413	90	0.090
Mille Lacs	1374	Northern Pike	4/23/2004	6/17/2004	22.6	1200	M	634	142	0.142
Mille Lacs	1364	Northern Pike	4/23/2004	6/17/2004	25.9	1900	M	718	155	0.155
Mille Lacs	1366	Northern Pike	4/23/2004	6/17/2004	26.2	2230	F	604	125	0.125
Mille Lacs	1381	Northern Pike	4/23/2004	6/17/2004	39.0	8130	F	889	199	0.199
Mille Lacs	1368	Northern Pike	4/23/2004	6/17/2004	39.0	5720	F	807	163	0.163
Mille Lacs	1373	Northern Pike	4/23/2004	6/17/2004	31.2	3210	F	635	149	0.149
Mille Lacs	1371	Northern Pike	4/23/2004	6/17/2004	33.6	3860	M	1589	345	0.345
Mille Lacs	1369	Northern Pike	4/23/2004	6/17/2004	33.4	3740	M	1585	321	0.321
Mille Lacs	1383	Northern Pike	4/23/2004	6/17/2004	30.9	3520	M	795	192	0.192
Mille Lacs	1379	Northern Pike	4/23/2004	6/17/2004	37.8	5530	F	2710	596	0.596

Lake	Tag number	Species	Date Collected	Date of Fish Dissection	Fresh Length (in)	Fish Weight+ Tag (grams)	Sex	Hg Conc Dry Wt (ng/g)	Hg Conc Wet Wt (ng/g)	Hg Conc Wet Wt (ug/g)
Mille Lacs	1363	Northern Pike	4/23/2004	6/17/2004	36.7	6490	F	1307	279	0.279
Mille Lacs	1376	Northern Pike	4/23/2004	6/17/2004	37.3	7060	F	728	161	0.161
Franklin	08862	Northern Pike	6/22/2004	2/9/2005	28.6	2030	F	1053	213	0.213
Franklin	08861	Northern Pike	6/22/2004	2/9/2005	21.5	970	F	545	113	0.113
Franklin	08873	Northern Pike	6/22/2004	2/9/2005	15.8	400	F	340	69	0.069
Franklin	08866	Northern Pike	6/22/2004	2/9/2005	25.7	1420	F	1714	336	0.336
Franklin	08871	Northern Pike	6/22/2004	2/9/2005	22.4	930	M	1516	291	0.291
Franklin	08867	Northern Pike	6/22/2004	2/9/2005	18.7	640	F	380	80	0.080
Franklin	09612	Northern Pike	6/22/2004	2/9/2005	20.7	750	M	849	150	0.150
Franklin	09503	Northern Pike	6/23/2004	2/9/2005	24.1	1240	F	657	142	0.142
Franklin	10766	Northern Pike	6/23/2004	2/9/2005	26.8	1710	F	822	170	0.170
Franklin	09610	Northern Pike	6/23/2004	2/9/2005	22.0	930	F	662	135	0.135
Franklin	09509	Northern Pike	6/23/2004	2/9/2005	26.8	1540	F	1231	253	0.253
Franklin	09506	Northern Pike	6/23/2004	2/9/2005	16.2	390	U	330	66	0.066
Franklin	1836	Northern Pike	6/23/2004	2/9/2005	28.9	2070	F	1059	218	0.218
Franklin	1833	Northern Pike	6/23/2004	2/9/2005	28.3	1940	F	1216	260	0.260
Franklin	1839	Northern Pike	6/23/2004	2/9/2005	21.0	810	M	684	144	0.144
Franklin	00045	Northern Pike	6/23/2004	2/9/2005	17.0	470	M	304	62	0.062
Franklin	08665	Northern Pike	6/23/2004	2/9/2005	13.0	200	M	238	48	0.048