

# GREAT LAKES FISHERY COMMISSION

## 2000 Project Completion Report<sup>1</sup>

### Contaminant Burdens in Great Lakes Larval Sea Lamprey

#### A 1999 Spatial Survey

by:

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# **CONTAMINANT BURDENS IN GREAT LAKES LARVAL SEA LAMPREY**

## **A 1999 SPATIAL SURVEY**

### **PRELIMINARY REPORT**

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## 1999 Spatial Survey

### ABSTRACT

Samples of larval sea lamprey and transformers collected from 10 streams flowing into Lakes Superior, Huron and Ontario were analysed for total body burdens of mercury. A subset of the samples was analysed for methyl mercury content. The highest concentrations of total mercury were found in individuals captured in a stream from the Lake Ontario basin. The mean value of mercury concentrations in larval lamprey collected from 6 Lake Superior basin streams was marginally greater (0.354 vs 0.349  $\mu\text{g/g}$ ) than the mean concentration in samples from the 3 Lake Ontario basin streams. In the Lake Superior basin samples from the Bad River system (Upper Marengo, Potato and Brunsweller Rivers) had significantly greater mean total mercury concentrations (0.411 vs 0.286  $\mu\text{g/g}$ ) than samples from the 3 other Lake Superior basin sites (Nipigon R., Wolf R. and Davignon Creek). Transformers collected from the 3 Bad River system streams had mean total mercury body burdens that were not significantly different (0.426 vs 0.411  $\mu\text{g/g}$ ) than those detected in larval lamprey from the same streams. Methyl mercury levels represented from 56 to > 90% of the total mercury levels in larvae from all sites. In the Lake Superior basin, where a larger number of methyl mercury analyses were conducted, larval lamprey had a higher % methyl mercury content (75.93 Vs 67.43 %) than that found in transformers, all of which were collected in the Bad River system. PCB and organochlorine pesticide levels were near the 0.002  $\mu\text{g/g}$  detection limit for larval lamprey based on an analysis of limited number of samples from the Lake Superior basin sites. Similarly toxaphene levels were consistently < 0.01  $\mu\text{g/g}$ , the limit of detection, in 10 samples of whole lamprey larvae collected from 2 Lake Superior streams.

### MATERIALS AND METHODS

From August through October, 1999 samples of larval sea lamprey were collected from 7 sites in the Lake Ontario, Lake Huron and Lake Superior (Fig.1). In September 1998 samples of both larval lamprey and transformers were collected from 3 streams within the Bad River system (Upper Marengo, Potato and Brunsweller Rivers) which flows into the southwestern portion of Lake

Superior. All larval lamprey and transformer samples were frozen immediately after collection and stored at  $-20^{\circ}\text{C}$  in contaminant free (FDA approved) plastic bags until processing. Larval lamprey were processed as composites. The number of individuals utilized to produce each composite sample was controlled by the final sample weight required for analysis. Composite samples ranged in size from  $< 5$  individuals to  $> 100$ . The ideal composites sample was large enough to provide a 15 g sample for analysis plus a duplicate sample and if possible a 10g sample for storage in the Great Lakes Tissue Archive facility. In 50% of the cases all 3 types of samples were available for larval lamprey analytical samples while in  $< 10\%$  of the cases only a single sample was available. Because of the limited number of transformers collected at the 3 Bad River system sites, all 6 samples consisted only of an analytical sample and a duplicate. In the laboratory, measurements of individual total length and weight were recorded for each larvae or transformer and mean values were computed for each sample (Table 1). Individuals were cut into  $< 1$  cm sections. These sections were homogenized using a Polytron<sup>®</sup> (Brinkman Kinematica Homogenizer, Lucerne, Switzerland) tissue grinder and the homogenates were divided into subsamples. Processing equipment and associated glassware were washed with distilled water and rinsed with distilled in glass (DIG) pesticide grade solvents (Caledon Laboratories Georgetown, ON) prior to use. All tissue homogenates were placed in acetone and hexane rinsed flint glass jars, sealed with an aluminum foil lined, hexane rinsed cap and stored at  $-20^{\circ}\text{C}$  for less than six weeks prior to analysis.

### **TOTAL MERCURY DETERMINATION**

One gram of lamprey tissue was weighed out into a volumetric flask. The tissue was then dissolved in 15 ml of sulfuric acid. Organic and inorganic mercury compounds, if present, were decomposed using nitric acid, potassium permanganate and potassium persulphate. Mercuric ions were reduced to the elemental state with stannous chloride. Mercury vapour is subsequently removed from solution by aeration and total mercury was determined via cold vapour, flameless atomic absorption using a LDC Milton-Roy elemental mercury monitor. The detection limit for total mercury is 0.01 mg/kg. A complete description of the extraction and analysis procedures for mercury is presented in Environment Canada (1994).

## **METHYL MERCURY DETERMINATION**

Methyl mercury was measured using a gas chromatograph with an electron capture detector (GC-ECD) as described in Wagemann et al. 1997. This procedure is an adaptation of the method described by Uthe et al. (1972) and involves a toluene extraction phase and analysis with a Varian model 3600 gas chromatograph with a <sup>13</sup>Ni ECD temperature programmable detector and a 5 m SPB-5 mega bore column (0.53 mm i.d.). The detector was maintained at 300°C at all times. The carrier gas was helium and the make up gas was hydrogen. The absolute detection limit for MeHg by ECD was 2pg (based on 3 X SD of blank analyses) or 10 to 80 ng/g Hg wet weight.

## **ORGANOCHLORINE COMPOUND ANALYSIS**

Organochlorine pesticides and total PCB analyses were performed using a Varian 3600 gas chromatograph (GC) with dual electron capture detectors (ECD). Organochlorine pesticide analysis required dual channel confirmation and a RTx-5 60m x .25mm x .25mm column was used with a RTx-1701 60m x .25mm x .25mm as the confirmation column.

Organochlorine compounds were quantified against an eight-point calibration curve, final results were corrected for recoveries and no blank corrections were performed. Total PCB's were quantified using a standard containing a 1:1:1 mixture of Aroclors 1242, 1254 and 1260 at a concentration of 500pg/ul. Total toxaphene was quantified against a 500pg/ul technical standard from Hercules Chemical. Twenty target peaks from the chromatogram were used in quantification. A detailed description of the extraction process and analytical method for total PCB's and OC's is presented in Huestis et. al. (1995).

## **RESULTS**

Table 1 presents a summary of the data associated with the 1999 sample collections. More than 50% of the larval lamprey samples came from 6 streams in the Lake Superior basin. Conversely only 5 composite samples were collected and analysed from the single Lake Huron site. Three sites along the north shore of Lake Ontario were surveyed. No samples were obtained from Lake Erie or Lake Michigan. Transformers were collected in 3 streams from the Lake Superior basin. These 3 streams, consisting of Upper Marengo, Potato and Brunsweller Rivers also had

simultaneous collections of larval lamprey. Therefore, a direct comparison of contaminant levels between these two life stages was possible. Although lamprey transformers from these sites were ~ 40% larger than the larval forms, total mercury levels were not significantly greater (0.438 vs 0.416  $\mu\text{g/g}$ ). Methyl mercury levels were identical (0.287 vs 0.290  $\mu\text{g/g}$ ) in both life stages sampled from the same three sites within the Bad River system.

On a basin wide basis, there were differences between the 3 lake systems surveyed with respect to mercury burdens in larval lamprey. The single Lake Huron site, with 5 samples analysed, generated the lowest mean lake specific total mercury value (0.292  $\mu\text{g/g}$ ) while the mean of the 3 Lake Ontario sites was equivalent to the mean of the 6 Lake Superior sites (0.340 vs 0.355  $\mu\text{g/g}$ ). The Lake Superior system had the largest range of the three lake surveyed with a minimum and maximum sample range of from 0.206 to 0.689  $\mu\text{g/g}$  total mercury in larval samples collected from 6 sites. Lake Ontario had a within lake variation in total mercury levels in larvae with a ranging from 0.186 to 0.513  $\mu\text{g/g}$  across the 3 sites surveyed.

In the Lake Superior basin samples from the Bad River system (Upper Marengo, Potato and Brunswellier Rivers) had significantly greater mean total mercury concentrations (0.411 vs 0.286  $\mu\text{g/g}$ ) than samples from the 3 other Lake Superior basin sites (Nipigon R., Wolf R. and Davignon Creek). Transformers collected from the 3 Bad River system streams had mean total mercury body burdens that were not significantly different (0.426 vs 0.411  $\mu\text{g/g}$ ) than those detected in larval lamprey from the same streams. Methyl mercury levels represented from 56 to > 90% of the total mercury levels in larvae from all sites. In the Lake Superior basin, where a larger number of methyl mercury analyses were conducted, larval lamprey had a higher % methyl mercury content (75.93 Vs 67.43 %) than that found in transformers, all of which were collected in the Bad River system.

Previous analyses of total mercury and methyl mercury levels in adult lamprey indicated a significantly higher percentage of methyl mercury in these types of samples. In adult lamprey, methyl mercury levels represented 96.8% of the total mercury levels measured (Whittle et al.). Depending on the analytical methods used, methyl mercury levels measured in fish tissues usually represents from 70 to < 90% of the total mercury concentration detected (Bloom, 1992, Kamps et al., 1972). Therefore, the mean methyl mercury content of larval lamprey more closely represents that found in fish and the larval levels are significantly less than those detected in adult lamprey. The

explanation for the exceedingly high methyl mercury levels in adult lamprey relates to the fact that during their parasitic phase, lamprey feed exclusively on a highly proteinaceous diet of blood and body fluids of the host salmonids. The mercuric ion readily binds to this material (Spacie et al., 1995) and thus the lamprey are feeding on a diet highly contaminated with methyl mercury.

Analysis of total mercury in adult lamprey collected in 1998 from streams in the same lake basins showed a significantly different spatial pattern. Table 3 provides a comparison of adult vs larval total mercury levels from Lakes Superior, Huron and Ontario. For adults, total mercury levels in Lake Superior were >50% higher than those in samples from Lake Ontario streams. Mean total mercury burdens in Lake Huron adult lamprey were > 50% less than those detected in the adults from the Lake Ontario sites. Larval lamprey did not display this interlake pattern. Lake Superior and Lake Ontario mean total mercury levels were not significantly different. Lake Huron levels were significantly less than those for larval samples from the Lake Superior basin.

Although the larval lamprey were significantly less contaminated than the adult lamprey in each of the 3 lakes surveyed the total mercury levels were greater than those detected in whole adult lake trout from the same 3 lake systems. Total mercury concentrations in whole lake trout from Lakes Superior, Huron and Ontario were 0.16, 0.07 and 0.14  $\mu\text{g/g}$  respectively (MacEachen et al.). Thus the spatial pattern of mercury contamination in larval lamprey more closely reflects that of body burdens in whole adult lake trout, but the larval lamprey levels are consistently greater than concentrations in the top predator lake trout.

PCB and organochlorine pesticide levels were near the 0.002  $\mu\text{g/g}$  detection limit for larval lamprey based on an analysis of limited number of samples from the Lake Superior basin sites. The only contaminant consistently detected in all samples was the DDT primary metabolite pp' DDE which was at concentrations between 0.002 and 0.005  $\mu\text{g/g}$  levels in 9 of the 10 larval lamprey samples analysed from the Wolf and Nipigon River sites on Lake Superior. An exception was a single Nipigon River sample with a pp'DDE concentration of 0.014  $\mu\text{g/g}$  whole animal concentration. The only other organochlorine pesticides consistently detected in these samples were heptachlor epoxide (B) and dieldrin. Only the Nipigon River samples had detectable levels of these two pesticides. Toxaphene levels were consistently < 0.01  $\mu\text{g/g}$ , the limit of detection, in 9 samples of whole lamprey larvae collected from the same 2 Lake Superior streams. A single sample from the

Wolf River site had a concentration of 0.064 µg/g toxaphene. As a result of the very low level of organochlorine compounds detected in these samples from Lake Superior, it was decided to defer any further organochlorine analyses on samples from Lake Huron and Lake Ontario. These samples were archived for possible future analyses.

## **CONCLUSIONS**

Larval lamprey within the Great Lakes basin provide a lake specific indicator of mercury contamination. The spatial pattern of total mercury accumulation is similar to that previously determined for the top predator lake trout. Their total body burdens consistently exceeded those measured in lake trout. The survey did reveal the slightly higher levels of mercury consistently detected in larval lamprey from the Bad River system in the Lake Superior basin. Methyl mercury to total mercury proportions in larval lamprey were similar to those measure in fish tissues but significantly less than those measured in adult parasitic phase lamprey. This difference is attributed to the contaminated diet of adult lamprey. Although lamprey transformers represent a latter life stage than larval lamprey, their level of total mercury was not significantly different than that detected in the larval forms analyzed from identical stream systems.

The level of organochlorine contaminants measured in a subsample of larval lamprey from Lake Superior streams was very low. Most contaminants were at or near the detection limit of the analytical methodology. There were some minor site to site differences in contamination for samples from Lake Superior basin streams. Samples were retained for future possible analysis.

## **RECOMMENDATIONS FOR FUTURE STUDIES**

1. Analysis of larval lamprey from both Lake Michigan and Lake Erie streams.
2. Expansion of the number of Lake Huron sites assessed.
3. Reassessment of the Bad River system for mercury contamination.



## **ACKNOWLEDGEMENTS**

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FIGURE 1.

# 1999 Lamprey Larvae Collection Sites

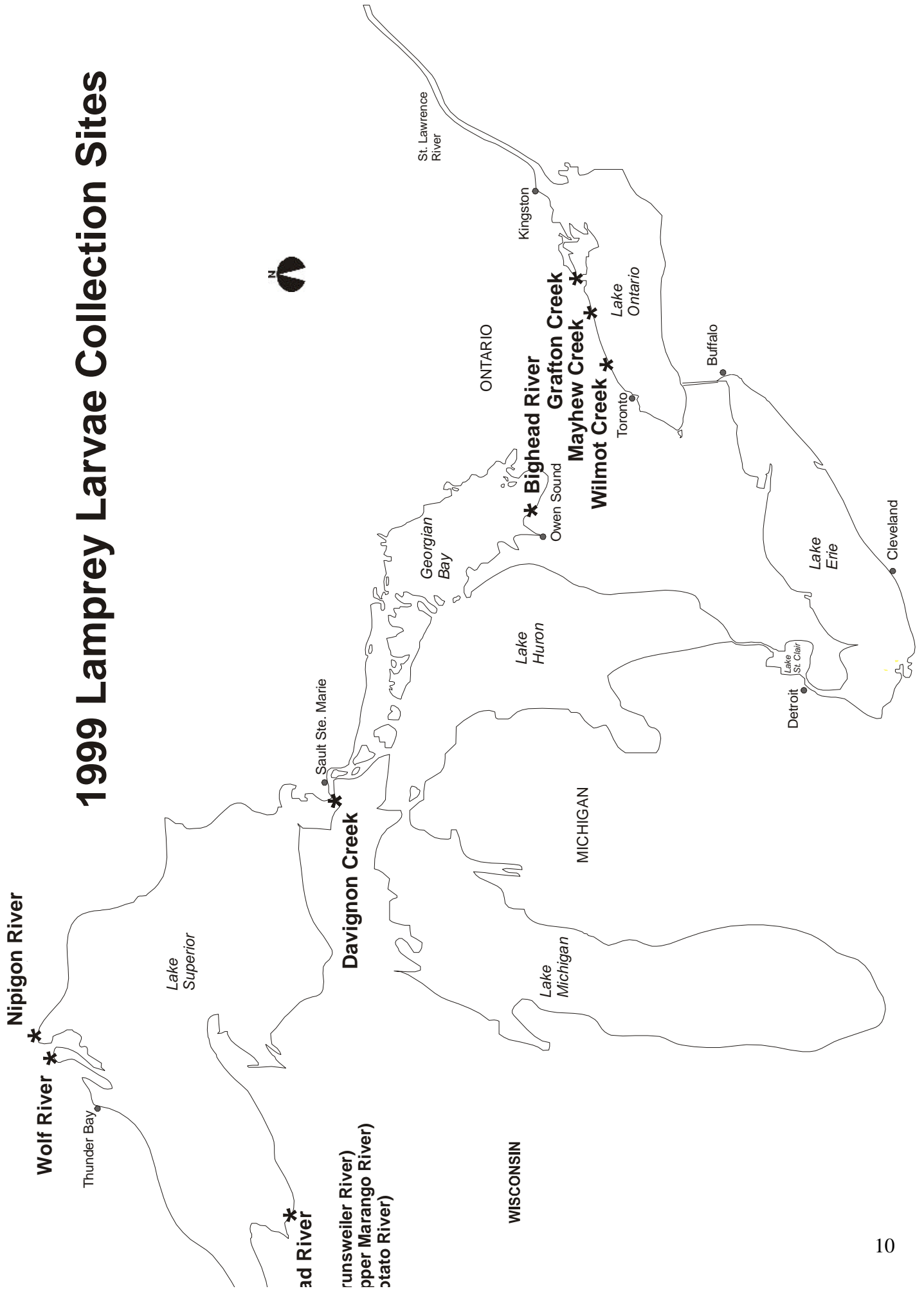


TABLE 1

## GREAT LAKES SEA LAMPREY LARVAE CONTAMINANTS DATA

CSP Sample #	Location	Site	Life Stage	Collection Date (dd,mm,yy)	Total Length (mm)	Wet Weight (g)	Total Hg (ug/g)	MeHg (ug/g)	%MeHg: Hg
16576	Lake Ontario	Wilmot Creek	larvae	23-Aug-99	7.5	1.0	0.322	---	---
16579	Lake Ontario	Grafton Creek	larvae	24-Aug-99	13.4	3.8	0.513	0.414	80.708
16580	Lake Ontario	Mayhew Creek	larvae	24-Aug-99	10.0	3.0	0.186	---	---
16571	Lake Huron	Big Head River	larvae	05-Oct-99	7.0	1.1	0.285	---	---
16572	Lake Huron	Big Head River	larvae	05-Oct-99	9.0	2.6	0.361	0.257	71.097
16573	Lake Huron	Big Head River	larvae	05-Oct-99	7.0	1.7	0.261	---	---
16574	Lake Huron	Big Head River	larvae	05-Oct-99	8.0	2.5	0.248	---	---
16575	Lake Huron	Big Head River	larvae	05-Oct-99	7.5	2.6	0.303	---	---
16551	Lake Superior	Wolf River	larvae	29-Aug-99	6.5	0.65	0.219	---	---
16552	Lake Superior	Wolf River	larvae	30-Aug-99	7.25	0.8	0.222	---	---
16553	Lake Superior	Wolf River	larvae	31-Aug-99	6.25	1.05	0.211	0.176	83.412
16554	Lake Superior	Wolf River	larvae	01-Sep-99	6.5	0.6	0.206	---	---
16555	Lake Superior	Wolf River	larvae	02-Sep-99	7	0.6	0.227	---	---
16556	Lake Superior	Wolf River	larvae	03-Sep-99	5.5	0.65	0.270	---	---
16557	Lake Superior	Nipigon River	larvae	30-Aug-99	10.5	2.7	0.260	---	---
16558	Lake Superior	Nipigon River	larvae	30-Aug-99	7.5	2.55	0.209	---	---
16559	Lake Superior	Nipigon River	larvae	30-Aug-99	9.5	1.55	0.409	---	---
16560	Lake Superior	Nipigon River	larvae	30-Aug-99	9	3.45	0.257	0.16	62.373
16568	Lake Superior	Nipigon River	larvae	30-Aug-99	8.5	2.8	0.266	---	---
16569	Lake Superior	Nipigon River	larvae	30-Aug-99	8.5	2.9	0.210	---	---
16570	Lake Superior	Nipigon River	larvae	30-Aug-99	7.3	1.5	0.217	---	---
16577	Lake Superior	Davignon Creek	larvae	04-Oct-99	6.0	1.3	0.522	---	---
16578	Lake Superior	Davignon Creek	larvae	04-Oct-99	7.0	2.1	0.222	0.183	82.432
16843	Lake Superior	Bad River (Brunswweiler River)	larvae	21-Sep-98	6.5	0.3	0.288	---	---
16844	Lake Superior	Bad River (Brunswweiler River)	larvae	21-Sep-98	10.0	1.8	0.437	---	---
16845	Lake Superior	Bad River (Brunswweiler River)	larvae	21-Sep-98	13.5	4.8	0.466	---	---
16846	Lake Superior	Bad River (Brunswweiler River)	larvae	21-Sep-98	13.5	3.9	0.602	---	---
16847	Lake Superior	Bad River (Brunswweiler River)	larvae	21-Sep-98	13.5	5.3	0.429	---	---
16848	Lake Superior	Bad River (Brunswweiler River)	larvae	21-Sep-98	16.5	6.8	0.514	0.288	56.022
16849	Lake Superior	Bad River (Brunswweiler River)	larvae	21-Sep-98	16.5	5.8	0.491	0.376	76.631
16850	Lake Superior	Bad River (Brunswweiler River)	larvae	21-Sep-98	16.5	5.3	0.434	---	---
16851	Lake Superior	Bad River (Upper Marengo River)	larvae	21-Sep-98	10.0	2.1	0.336	---	---
16852	Lake Superior	Bad River (Upper Marengo River)	larvae	21-Sep-98	6.5	0.1	0.497	---	---
16853	Lake Superior	Bad River (Upper Marengo River)	larvae	21-Sep-98	10.5	1.0	0.483	---	---
16854	Lake Superior	Bad River (Upper Marengo River)	larvae	21-Sep-98	13.4	3.8	0.689	---	---
16855	Lake Superior	Bad River (Upper Marengo River)	larvae	21-Sep-98	13.5	3.4	0.421	---	---
16856	Lake Superior	Bad River (Upper Marengo River)	larvae	21-Sep-98	13.5	3.8	0.413	---	---
16857	Lake Superior	Bad River (Upper Marengo River)	larvae	21-Sep-98	16.5	5.6	0.357	0.298	83.45
16858	Lake Superior	Bad River (Upper Marengo River)	larvae	21-Sep-98	16.5	5.0	0.539	0.321	59.484
16895	Lake Superior	Bad River (Potato River)	larvae	22-Sep-98	6.0	0.4	0.296	---	---
16896	Lake Superior	Bad River (Potato River)	larvae	22-Sep-98	10.0	2.2	0.341	---	---
16897	Lake Superior	Bad River (Potato River)	larvae	22-Sep-98	10.0	1.9	0.351	---	---
16898	Lake Superior	Bad River (Potato River)	larvae	22-Sep-98	13.5	3.7	0.340	0.310	91.237
16899	Lake Superior	Bad River (Potato River)	larvae	22-Sep-98	16.5	5.0	0.284	0.251	88.325
16902	Lake Superior	Bad River (Potato River)	larvae	22-Sep-98	6.5	0.2	0.300	---	---
16903	Lake Superior	Bad River (Potato River)	larvae	22-Sep-98	10.0	1.9	0.248	---	---
16891	Lake Superior	Bad River (Upper Marengo River)	transformers	21-Sep-98	15.0	6.2	0.438	0.286	65.165
16892	Lake Superior	Bad River (Upper Marengo River)	transformers	21-Sep-98	17.0	6.5	0.475	0.279	58.657
16893	Lake Superior	Bad River (Brunswweiler River)	transformers	21-Sep-98	18.0	9.5	0.393	0.285	72.489
16894	Lake Superior	Bad River (Brunswweiler River)	transformers	21-Sep-98	17.4	9.2	0.391	0.307	78.523
16900	Lake Superior	Bad River (Potato River)	transformers	22-Sep-98	17.0	8.0	0.485	0.345	71.065
16901	Lake Superior	Bad River (Potato River)	transformers	22-Sep-98	18.0	7.4	0.376	0.221	58.708

TABLE 2

## MEAN MERCURY LEVELS IN GREAT LAKES LARVAL LAMPREY

Lake	Site	Life Stage <sup>1</sup>	Collection Date	Length (mm) Mean ? S.E.	Weight (g) Mean ? S.E.	Hg (ug/g wet weight) Mean ? S.E.
Lake Ontario N = 1	Wilmot Creek	larvae	23-Aug-99	7.500 ---	1.000 ---	0.322 ---
Lake Ontario N = 1	Grafton Creek	larvae	24-Aug-99	13.400 ---	3.800 ---	0.513 ---
Lake Ontario N = 1	Mayhew Creek	larvae	24-Aug-99	10.000 ---	3.000 ---	0.186 ---
Lake Huron N = 5	Big Head River	larvae	5-Oct-99	7.700 0.374	2.100 0.302	0.292 0.020
Lake Superior N = 6	Wolf River	larvae	29-Aug-99	6.500 0.250	0.726 0.071	0.226 0.009
Lake Superior N = 7	Nipigon River	larvae	30-Aug-99	8.686 0.421	2.493 0.272	0.261 0.026
Lake Superior N = 2	Davignon Creek	larvae	4-Oct-99	6.500 0.500	1.700 0.400	0.372 0.150
Lake Superior N = 8	Bad River Brunswailer River	larvae	21-Sep-98	13.313 1.253	4.250 0.769	0.458 0.032
Lake Superior N = 8	Bad River Upper Marengo River	larvae	21-Sep-98	12.550 1.203	3.100 0.673	0.467 0.040
Lake Superior N = 7	Bad River Potato River	larvae	22-Sep-98	10.357 1.396	2.186 0.646	0.309 0.014
Lake Superior N = 2	Bad River Upper Marengo River	transformers	21-Sep-98	16.000 1.000	6.350 0.150	0.457 0.018
Lake Superior N = 2	Bad River Brunswailer River	transformers	21-Sep-98	17.700 0.300	9.350 0.150	0.392 0.011
Lake Superior N = 2	Bad River Potato River	transformers	22-Sep-98	10.500 6.500	7.700 0.300	0.430 0.054

<sup>1</sup> All Samples Analyzed As Composites of Whole Animals

**TABLE 3**

**TOTAL MERCURY BURDENS IN GREAT LAKES LARVAL & ADULT SEA LAMPREY <sup>(1)</sup>**  
**(Mean ? S.E. as ? g/g wet weight)**

<b>Lake</b>	<b>Adult</b>	<b>Larvae</b>
<b>SUPERIOR</b>	2.697 ? 0.179	0.354 ? 0.017
<b>HURON</b>	0.479 ? 0.111	0.279 ? 0.020
<b>ONTARIO</b>	1.096 ? 0.137	0.340 ? 0.010

**(1) Whole Lamprey**