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**APPENDIX B**  
**1996 TERN EGG COLLECTION AND CHEMICAL ANALYSIS PROCEDURES**

**Standard Operating Procedure for Collection, Transport, and Storage of Tern Eggs**

**Results of Chemical Analysis of Tern Eggs**

**Chemical Analysis Technical Procedures**

**Data Validation Report**

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# STANDARD OPERATING PROCEDURE FOR THE COLLECTION, TRANSPORT, AND STORAGE OF TERN EGGS FROM GREEN BAY, WISCONSIN

## 1. INTRODUCTION AND STUDY OBJECTIVES

This Standard Operating Procedure (SOP) contains the objectives, methods, and approaches for the collection, transport, and storage of Common tern (*Sterna hirundo*), Forster's tern (*Sterna forsteri*), and Caspian tern (*Sterna caspia*) eggs to be collected from Green Bay, Wisconsin, for the Fox River and Green Bay Natural Resource Damage Assessment (NRDA). The collected eggs will be analyzed for contaminants by an analytical laboratory. A subsequent SOP will describe the laboratory analytical methods that will be employed.

The objective of the study is to:

- ▶ Collect eggs of the tern species listed above from colonies in the Lower Fox River and Green Bay to provide comparisons between current and historical egg contaminant concentrations.

Tern eggs will be collected during the 1996 nesting season (and, if necessary, during the 1997 nesting season) and will be analyzed for PCBs (congener-specific analyses), and potentially other contaminants. The field team leader for the egg collection will be Dr. Hector Galbraith.

## 2. FIELD PROCEDURES

### 2.1 TERN COLONY LOCATION

Suitable tern nesting colonies will be located by U.S. Fish and Wildlife Service (USFWS) personnel during the early part of the 1996 (and, if necessary, 1997) nesting season. Caspian tern eggs will be collected from the known breeding colony on Gravelly Island, Green Bay. For Foster's and common terns, the egg collections will be made from Kidney Island in the Lower Fox River. If no terns of either species nest on Kidney Island, or the numbers of nesting birds are too low to provide the required sample sizes (see below), the west shore of Green Bay will be searched for nesting colonies, and eggs will be collected from those colonies closest to the mouth of the Lower Fox River.

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## 2.2 EGG COLLECTION

Eggs from at least 6 nests will be collected for each species. If the colony contains more than 6 nests, each nest will be located and uniquely numbered. A random number generator will then be used to identify 6 nests. Up to 2 eggs (depending on the clutch size) will be collected from each of the selected nests. If no colony of 6 or more nests is found, a number of colonies will be combined into a hypothetical colony, the nests numbered, and study nests randomly chosen.

Each collected egg will be given a unique numerical identifier in the field. This number will be written on the egg in pencil. All identification numbers will be recorded in the field logbook. The identification system for eggs samples collected for contaminant analyses consists of the following code:

**TE-XX-Y-AB**

where:

- ▶ **TE** is a two-letter code designating the tern egg collection effort.
- ▶ **XX** is a unique two-letter code designating the colony location
- ▶ **Y** is a tern species identifier (A = common, B = Forster's, C = Caspian)
- ▶ **##** is a unique two-number code designating the nest number. Nests will be numbered starting at "01."

## 2.3 FIELD DOCUMENTATION

The field team will document its sampling activities and field measurements in a dedicated, paginated, bound field logbook. Sampling locations will be clearly identified on photocopies of appropriate topographical maps and described in the field notebook. Entries in the field notebook and map marking will be done with waterproof ink, and corrections will be made with a single line through the error accompanied by the correction date and corrector's initials. The field team leader will be responsible for maintenance and proper archiving of these field notebooks.

The following information will be recorded in the field logbooks:

- ▶ site and project name
  - ▶ each sampler's name and professional affiliation
  - ▶ approximate numbers of nests in each colony
  - ▶ clutch size in each selected nest
  - ▶ date and time of egg collection, field activity, or field measurement
  - ▶ identification numbers of samples collected
  - ▶ number and type of samples collected
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- ▶ any difficulties encountered or necessary deviations from this SOP
- ▶ any other pertinent field observations.

Maps will be marked with a sampling location code, e.g., PE for Peshtigo River, written within a circle. The field notebook page number corresponding to each sampling location will be marked adjacent to the sampling location circle. Photographs will also be taken of each colony.

Upon completion of each day's field activities, the notes will be reviewed by the field recorder and sampler and any necessary corrections made. The field recorder will sign and date each page.

## 2.4 PROCESSING AND STORAGE OF EGGS

The field team leader or a designated representative will transport the eggs to the USFWS laboratory in Green Bay. Immediately on returning from the field to the laboratory, the eggs will be measured and their contents transferred to chemically clean glass jars. Egg measurements will be made using a Vernier caliper and an electronic balance and will include:

- ▶ length and breadth (to the closest 0.1 mm).
- ▶ weight (to the closest 0.1g).
- ▶ egg volume using water displacement in a gravimetric flask

These measurements will be recorded in the field notebook.

After the above measurements are taken, the contents of each egg will be transferred to a pre-labeled, tared, pre-cleaned and certified glass container and the jar plus egg contents weighed to the closest 0.1g. The jar tare weights and the jar plus contents weights will be recorded in the field log book. The jars will be stored in a freezer to await shipment to the analytical laboratory.

The tern egg shells will be labeled with the egg identifier, allowed to air dry, then stored in a sealed egg box in a dry area within the USFWS field office at Green Bay.

## 2.5 CHAIN OF CUSTODY

The chain of custody will start when eggs are collected from the nests. Each egg will be given a unique numerical identifier in the field. This number will be written on the egg in pencil. Once identified in this way, the eggs collected during each sampling event will be placed in a communal egg container under the custody of Dr. Hector Galbraith or a designated stand-in. On returning to the laboratory, the contents of each egg will be transferred to separate chemically clean glass jars. Each of these jars will be labeled with the appropriate sample identifier. The jars will be stored frozen in one or more shipping containers which will be sealed with custody seals (to detect

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unauthorized tampering with samples after sample collection until the time of use or analysis), and contain chain of custody forms with the following information, as appropriate:

- ▶ project name
- ▶ egg identifiers (unique for each sample)
- ▶ name and signature of field recorder
- ▶ date and time of beginning of sample collection
- ▶ chain of custody seal number
- ▶ signatures of persons involved in the chain of possession
- ▶ inclusive dates and times of possession
- ▶ method and date of sample shipment.

At the appropriate time, the entire sealed container(s) will be shipped to the analytical laboratory.

The field recorder is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. A sample is in the custody of an individual if any of the following occur:

- ▶ The sample is in the individual's possession.
- ▶ The sample is within view after being in possession.
- ▶ The sample is in a locked or sealed container that prevents tampering after being in possession.
- ▶ The sample is in a designated secure area.

Every transfer of custody will be noted with the date and time of transfer and signed for on the chain of custody record. The number of custody transfers will be kept to a minimum.

## **2.7 FIELD EQUIPMENT**

The following list of equipment will be required in the field:

- ▶ SOPs (one copy for each team member)
  - ▶ waders/hip boots (all crew members)
  - ▶ field log books
  - ▶ marking pens and pencils
  - ▶ labels and labeling tape
  - ▶ chain of custody forms and seals
  - ▶ an egg box for sample storage and transport
  - ▶ kimwipes
  - ▶ camera
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## **2.8 DEVIATIONS FROM THIS SOP**

If field conditions necessitate any deviations from this SOP the Field Team Leader will document them in the field note book and in an addendum to this SOP.

## ADDENDUM TO TERN EGG COLLECTION SOP

During the course of fieldwork four changes were made to the egg collection method. The decision to make these changes was made by the field team leader. The changes are:

**1) Sampling Methods in Forster's Tern Colony.** The method described in Section 2.2 of this SOP was changed. The method described in Section 2.2 was developed under the assumption that any Forster's tern colonies found would have relatively few widely dispersed pairs. In fact, the Kidney Island Forster's tern colony comprised about 100 pairs densely settled in a relatively small area. Also, the Kidney Island terns nested immediately adjacent to several hundred pairs of ring-billed gulls (*Larus delawarensis*). Any attempt to number each of the nests and to randomly select study nests, as described in Section 2.2, would have resulted in prolonged disturbance to the birds, with the risk of predation of unguarded eggs by gulls. For these reasons, the following method was adopted:

- ▶ the Forster's tern colony was delineated and the numbers of nests counted. Two colonies were found: the main colony comprised 65 nests distributed in an ovoid approximately 20 meters by 60 meters. Another 20 to 30 pairs of terns were nesting in a smaller colony 50 meters to the east of the main colony.
- ▶ The main colony was walked through from south to north (along the colonies 60 meter axis) and a single egg was collected from each 6th nest. This provided a sample of 10 eggs.

**2) One Egg Was Collected From Each Nest.** The collection permit provided by the State of Wisconsin Department of Natural Resources allowed the collection of 10 Forster's and common tern eggs only. The field team leader decided that, in the interests of characterizing the colonies most fully, 10 nest should be sampled. This entailed the collection of one egg from each nest, not the maximum of 2 described in section 2.2.

**3) Common Tern Egg Collection.** At the time of the collection of the Forster's tern eggs, no common terns were nesting on Kidney Island. However, a visit two weeks later revealed that common terns had by then established themselves. A total of 15 nests were found. One egg was collected from each of 10 randomly chosen nests.

**4) No Caspian Tern Eggs Were Collected.** No attempt was made to locate and collect the eggs of Caspian terns.

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## Results of chemical analysis of tern eggs

The column "field.id" represents the identification number of each egg collected. 1996 is the year of collection. "KI" denotes the collection site of Kidney Island, and the last two values (common tern) or three values (Forster's tern) are the sample number (i.e., B01, B02).

The "analyte" column identifies the PCB congener for which a value is given (i.e., c.1.ppb.wwt means PCB congener 1, measured in parts per billion wet weight).

All values of 0 denote values below the detection limit.

Batch Matrix Tern Eggs  
 Region, Kidney Island, Green Bay  
 Reporting Unit: ng/q, wet weight

Client Reporting Sample ID	96KICT01_rep.1 Common Term	96KICT03_rep.1 Common Term	96KICT03_rep.2 Common Term	96KICT05_rep.1 Common Term	96KICT07_rep.1 Common Term	96KICT09_rep.1 Common Term	96KICT10_rep.1 Common Term	96KICT10_rep.2 Common Term	TEKIB06_rep.1 Forster's Tern
Sample Type:	5.4	7.7	7.7	9.4	7.5	6.3	7.7	7.7	7.8
Sample Lipid Content (%):	73.8	79.1	79.1	78.5	72.5	74.7	70.4	70.4	78.4
Sample Moisture Content (%):									
Congener Numbers									
c.001	0.0 U								
c.003	0.0 U								
c.004+010	0.8 U	1.1 U	0.0 U	1.2 U	0.0 U				
c.005	1.0 J	0.3 J	0.5 J	0.5 J	0.5 J	0.0 U	0.0 U	0.0 U	5.2 U
c.007.009	0.7 J	0.7 J	0.0 U	3.6 J					
c.008.005	0.0 U	60.8	0.0 U	4.4 J					
c.012.013	4.1 J	1.7 J	1.7 J	1.7 J	3.2 J	3.1 J	1.9 J	0.0 U	23.6
c.016.032	2.3 J	1.0 J	1.0 J	0.5 J	0.0 U	0.9 J	0.0 U	0.0 U	8.4
c.017.015	1.5 J	0.0 U	0.0 U	0.0 U	0.0 U	1.1 J	0.0 U	0.0 U	4.8 J
c.018	0.0 U	5.5 J							
c.019	0.0 U	0.2 J	0.2 J	0.0 U					
c.021	10.2	5.5 J	5.5 J	3.7 J	3.1 J	1.0 J	6.1 J	0.0 U	11.1
c.022	0.3 U	0.0 U	0.0 U	0.2 U	0.0 U	0.6 J	0.0 U	0.0 U	2.4 J
c.024+027	2.4 J	1.7 J	1.7 J	1.1 J	1.5 J	3.2 J	1.9 J	0.0 U	3.7 J
c.025	1.8 J	1.5 J	1.5 J	0.8 J	1.1 J	4.6 J	1.7 J	0.0 U	3.6 J
c.026	167.8	181.1	181.1	138.5	59.8	134.6	165.8	0.0 U	164.2
c.028	1.5 J	0.8 J	0.8 J	0.9 J	0.0 U	0.4 J	0.0 U	0.0 U	4.3 J
c.029	63.4	56.3	56.3	43.1	20.3	57.1	51.1	0.0 U	62.8
c.031	6.0 J	3.0 J	3.0 J	1.2 J	0.0 U	3.0 J	3.0 J	0.0 U	10.2
c.033.020	0.7 J	1.3	1.3	0.8 J	0.5 J	1.4 J	0.9	0.9	0.2 J
c.040	0.0 U	2.6 J	0.0 U	0.0 U	16.0				
c.041+064+071	0.0 U	8.6	2.6 J	0.0 U	134.0				
c.042+037	34.6	12.4	12.4	10.3	12.1	47.7	16.1	0.0 U	62.8
c.043	0.0 U	1.2 J							
c.044	3.2 J	0.8 J	0.8 J	1.0 J	6.5 J	23.3	1.8 J	0.0 U	130.8
c.045	0.0 U								
c.046	1.5 J	0.6 J	0.6 J	1.1 J	0.0 U	0.0 U	0.0 U	0.0 U	2.3 J
c.047+075	172.7	143.7	143.7	114.6	52.2	92.6	133.7	0.0 U	133.6
c.048	11.8	11.2	11.2	7.5	3.4 J	7.5 J	6.2 J	0.0 U	14.5
c.049	197.8	47.1	47.1	113.3	38.6	166.7	188.4	0.0 U	127.4
c.051	0.0 U								
c.052	88.0	20.4	20.4	36.4	37.2	169.8	26.0	0.0 U	335.2
c.053	162.2	130.8	130.8	96.5	54.7	124.8	105.0	0.0 U	3.9
c.056+060	0.7 U	0.8 U	0.8 U	0.4 U	1.2 U	2.8 U	0.2 U	0.0 U	104.5
c.059	50.8 J	26.4 J	26.4 J	48.3 J	7.2 J	41.9 J	29.2 J	0.0 U	9.3
c.063	543.0	481.5	481.5	390.2	183.8	306.3	413.8	0.0 U	26.2 J
c.065	229.2	166.1	166.1	143.7	86.2	191.1	150.8	0.0 U	336.0
c.070+076	355.0	246.8	246.8	210.7	89.0	167.6	253.3	0.0 U	147.5
c.074	8.9	10.4	10.4	7.7 J	3.9 J	8.0 J	7.1	0.0 U	211.5
c.077	1.0 J	0.7	0.7	1.4 J	0.8 J	1.2 J	0.9	0.0 U	2.3
c.081	25.3	12.1	12.1	17.8	12.9	41.3	12.1	0.0 U	1.2 J
c.082	3.4 J	2.1 J	2.1 J	2.9 J	13.5	5.7 J	3.4 J	0.0 U	35.5
c.083	25.0	7.7	7.7	20.0	26.5	40.2	6.6 J	0.0 U	70.9
c.084	77.1 J	42.7 J	42.7 J	58.5 J	19.8 J	102.1 J	117.0 J	0.0 U	874.3 J
c.085	87.4	18.5	18.5	37.1	29.6	88.9	65.9	0.0 U	46.9
c.087+115+081	1.5 J	1.2 J	1.2 J	1.2 J	2.1 J	1.5 J	0.5 J	0.0 U	1.3 J
c.089	44.0	23.0	23.0	27.8	17.8	33.8	21.0	0.0 U	44.8
c.091	9.0	3.2 J	3.2 J	14.8	3.7 J	8.2	3.3 J	0.0 U	59.6
c.092	12.5	107.9	107.9	4.7 J	7.6 J	23.5	26.6	0.0 U	113.6

Client Reporting Sample ID: Sample Type: Sample Lipid Content (%): Sample Moisture Content (%)	96KICT01_rep.1 Common Term	96KICT03_rep.1 Common Term	96KICT03_rep.2 Common Term	96KICT05_rep.1 Common Term	96KICT07_rep.1 Common Term	96KICT09_rep.1 Common Term	96KICT10_rep.1 Common Term	96KICT10_rep.2 Common Term	TEKIB06_rep.1 Forster's Term
c.097	41.4	15.9		21.7	213	73.6	17.5		75.6
c.099	524.2	321.4		310.2	198.8	236.5	398.2		391.4
c.100	4.3	3.7	J	2.4	1.6	3.3	3.2	J	5.3
c.101+090	396.8	152.0		229.2	155.2	286.9	260.8		346.5
c.105	176.9	113.8		119.5	108.0	121.6	127.9		118.1
c.107+147	10.3	8.0	J	26.4	17.2	15.6	22.0		20.9
c.110+077	259.1	155.7		181.6	175.6	258.0	188.5		221.7
c.114	16.0	10.4		11.9	12.2	13.4	12.7		12.5
c.116	698.6	389.3		399.8	351.4	359.7	486.8		421.3
c.119	20.5	14.4		12.6	6.3	8.6	12.9		14.0
c.124	5.5	4.4	J	3.0	4.1	6.4	2.2	J	2.8
c.126	2.0	1.5	J	1.3	0.6	0.9	1.2	J	0.6
c.128	110.5	54.6		62.7	61.5	53.3	75.8		77.5
c.129+126	3.4	1.4	J	1.7	1.6	5.0	1.4	J	7.0
c.130	25.3	14.8		13.3	13.3	21.0	15.1		38.4
c.131	3.6	2.6	J	3.2	3.0	6.6	1.4	J	12.6
c.132	141.4	32.7	J	30.0	54.4	39.6	269.0	J	42.2
c.134	0.0	0.0	U	0.0	0.0	0.0	0.0	U	0.0
c.135+144	6.5	0.9	J	1.4	3.5	2.7	4.5	J	22.0
c.136	0.4	0.0	U	0.0	0.0	0.0	0.2	J	5.1
c.137	40.7	16.6		21.7	23.0	19.1	25.7		27.4
c.138+150+163	824.3	394.8		481.1	474.1	346.9	555.3		528.8
c.141+179	4.6	4.0	J	2.7	7.8	7.3	3.7	J	2.6
c.146	146.2	83.2		70.2	85.4	86.2	82.4		125.4
c.149+123	151.7	55.0		96.6	106.8	140.8	78.6		163.4
c.151	3.1	1.1	J	2.1	4.0	6.5	1.1	J	22.1
c.153	1026.4	438.6		559.3	596.5	439.3	675.3		641.1
c.156	58.0	28.5		35.8	30.5	26.2	38.9		38.0
c.158	55.6	20.7		27.5	35.1	23.4	38.1		36.1
c.167	45.7	21.2		27.7	26.6	21.5	28.6		28.6
c.169	0.2	0.2	U	0.1	0.1	0.1	0.1	U	0.4
c.170+190	168.5	54.9	J	73.1	73.6	52.6	116.4	J	104.9
c.171+202	55.0	20.6		27.1	28.3	21.3	35.0		37.7
c.172	46.9	18.4		22.8	20.9	18.2	26.1		31.0
c.173	0.7	0.0	U	0.7	1.0	0.0	0.3	J	1.2
c.174	28.2	11.3		18.1	14.8	22.9	18.2		33.7
c.175	6.7	1.9	J	2.1	1.4	2.7	3.3	J	4.4
c.176	10.6	4.8	J	10.7	15.4	16.9	3.3	J	7.6
c.177	43.9	28.0		30.2	23.0	27.9	29.1		48.8
c.178	9.2	5.1	J	5.1	5.4	9.3	4.7	J	28.5
c.180	634.7	252.7		334.8	388.5	266.4	413.5		350.3
c.183	152.6	56.3		75.5	93.3	56.9	104.1		78.9
c.185	2.1	0.0	U	1.4	1.1	1.9	1.7	J	2.9
c.187+182	283.5	112.5		149.2	149.2	136.8	168.0		236.9
c.189	8.3	3.2	J	4.4	4.3	2.5	5.1	J	4.8
c.191	6.7	3.1	J	4.5	4.8	3.5	5.3	J	4.8
c.193	27.5	10.8		14.5	12.5	9.8	16.7		17.2
c.194	77.9	32.8		41.0	40.7	31.6	56.0		50.5
c.195+209	37.0	16.1	J	16.5	19.2	14.9	25.0	J	29.5
c.197	5.4	2.4	J	3.0	2.7	2.4	3.5	J	3.3
c.198	4.6	2.5	J	2.0	1.9	2.4	3.0	J	4.2
c.199	66.8	34.3		37.8	37.0	39.3	51.1		62.0
c.200	1.7	1.1	J	1.3	0.0	2.5	1.1	J	5.1
c.201+157	4.7	3.2	J	3.8	3.1	3.9	3.5	J	6.1
c.203+196	108.1	45.1		55.3	57.1	45.1	79.0		68.9
c.205	11.2	6.2	J	6.8	5.1	6.3	9.2	J	7.8
c.206	30.5	17.7		18.9	18.3	17.2	25.9		26.2
c.207	6.8	4.0	J	4.0	4.0	3.6	5.9	J	5.6
c.209	13.2	11.6	J	7.6	6.7	9.6	13.3	J	11.6
cond.sum	9010.9	4946.6		5244.7	4413.6	5390.3	6411.2		8091.7
comp.sum.ies95	8933.8	4903.9		5186.1	4393.8	5288.2	6294.2		7217.4

96KICT01

96KICT03

96KICT05

96KICT07

96KICT09

96KICT10

TEKIB06

Batch Matrix Reporting Unit

Client Reporting Sample ID:  
 Sample Type:  
 Sample Lipid Content (%):  
 Sample Moisture Content (%)

Region: Kidney Island, Green Bay  
 Term Eggs  
 ng/g, wet weight

TEKIB18\_rep.1  
 Forsler's Tern  
 8.5  
 77.3

Region: Kidney Island, Green Bay  
 Term Eggs  
 ng/g, wet weight

TEKIB18\_rep.2  
 Forsler's Tern  
 8.5  
 77.3

TEKIB24\_rep.1  
 Forsler's Tern  
 6.6  
 77.9

TEKIB30\_rep.1  
 Forsler's Tern  
 5.2  
 77.6

TEKIB48\_rep.1  
 Forsler's Tern  
 7.0  
 76.1

TEKIB48\_rep.2  
 Forsler's Tern  
 7.0  
 76.1

TEKIB60\_rep.1  
 Forsler's Tern  
 6.1  
 73.3

Congener Numbers

c.001	0.0	U										
c.003	0.0	U										
c.004+010	0.0	U										
c.006	0.0	U	3.6	U	1.0	U	1.4	U	1.4	U	2.1	U
c.007-009	0.0	U	1.2	J	0.4	J	0.3	J	0.3	J	0.4	J
c.008-006	0.0	U	2.8	J	0.0	U	0.0	U	0.0	U	1.1	J
c.012-013	0.0	U										
c.018-032	3.1	J	6.3	J	3.3	J	3.0	J	3.0	J	5.7	J
c.017-015	0.5	J	2.4	J	1.4	J	0.9	J	0.9	J	1.6	J
c.018	1.9	J	3.8	J	3.0	J	2.1	J	2.1	J	3.2	J
c.019	0.0	U										
c.021	0.0	U										
c.022	7.0	J	7.1	J	4.9	J	8.7	J	8.7	J	4.6	J
c.024+027	0.0	U	1.7	J	0.9	J	0.8	J	0.8	J	1.3	J
c.025	0.8	J	1.3	J	0.0	U	1.4	J	1.4	J	1.1	J
c.026	5.3	J	7.2	J	8.1	J	4.9	J	4.9	J	1.8	J
c.028	167.9	-	252.1	-	96.7	-	140.5	-	140.5	-	122.8	ok
c.029	0.0	U	1.2	J	0.0	U	0.0	U	0.0	U	0.0	U
c.031	48.3	-	73.1	-	32.1	-	36.6	-	36.6	-	36.5	ok
c.033-020	1.9	J	3.5	J	1.9	J	1.9	J	1.9	J	1.2	J
c.037	0.3	J	0.2	J	0.2	J	0.6	J	0.6	J	0.3	J
c.040	7.9	J	9.7	J	10.7	J	7.0	J	7.0	J	9.4	ok
c.041+064+071	3.6	J	4.5	J	0.0	U	0.0	U	0.0	U	0.0	U
c.042+037	39.5	-	40.6	-	34.9	-	28.0	-	28.0	-	31.2	ok
c.043	0.0	U										
c.044	78.2	-	101.0	-	82.1	-	64.5	-	64.5	-	86.3	ok
c.045	0.0	U										
c.046	0.0	U										
c.047+075	123.1	-	123.1	-	66.7	-	104.5	-	104.5	-	100.7	ok
c.048	8.2	J	36.0	-	7.1	J	7.8	J	7.8	J	6.3	J
c.049	71.7	-	91.0	-	98.0	-	56.7	-	56.7	-	75.8	ok
c.051	0.0	U										
c.052	269.2	-	379.3	-	200.1	-	258.2	-	258.2	-	239.3	ok
c.053	1.4	J	2.5	J	1.9	J	1.0	J	1.0	J	0.8	U
c.056+060	73.7	-	67.9	-	49.6	-	74.2	-	74.2	-	67.3	ok
c.059	4.5	U	5.3	J	5.5	J	3.3	U	3.3	U	3.9	U
c.068	36.9	J	21.2	J	17.9	J	31.8	J	31.8	J	30.2	J
c.065	311.5	-	404.9	-	194.2	-	318.4	-	318.4	-	298.8	ok
c.070+078	109.3	-	89.2	-	76.1	-	96.1	-	96.1	-	69.9	ok
c.074	180.1	-	232.2	-	115.0	-	171.4	-	171.4	-	165.1	ok
c.077	3.3	J	1.2	-	1.2	-	3.1	-	3.1	-	1.7	ok
c.081	1.3	J	0.7	J	0.5	J	0.5	J	0.5	J	0.6	J
c.082	23.0	-	15.6	-	17.8	-	16.1	-	16.1	-	14.9	ok
c.083	8.0	J	8.7	J	9.3	J	6.3	J	6.3	J	7.1	J
c.084	36.7	-	22.2	-	28.9	-	28.1	-	28.1	-	75.3	ok
c.085	283.2	J	0.0	J	85.4	J	31.4	J	31.4	J	48.2	J
c.087+115+081	28.2	-	31.0	-	36.7	-	25.8	-	25.8	-	31.3	ok
c.089	0.7	J	0.0	U								
c.091	27.6	-	27.2	-	23.4	-	23.8	-	23.8	-	34.3	ok
c.092	36.9	-	48.1	-	28.6	-	40.7	-	40.7	-	35.8	ok
c.095	46.4	-	66.7	-	49.2	-	57.8	-	57.8	-	47.0	ok

Client Reporting Sample ID:  
 Sample Type:  
 Sample Lipid Content (%):  
 Sample Moisture Content (%):

	TEKIB18_rep-1 Forster's Tern	TEKIB18_rep-2 Forster's Tern	TEKIB24_rep-1 Forster's Tern	TEKIB30_rep-1 Forster's Tern	TEKIB48_rep-1 Forster's Tern	TEKIB48_rep-2 Forster's Tern	TEKIB60_rep-1 Forster's Tern
c.097	60.5		55.9	41.5	52.7		43.2 ok
c.099	246.4		299.4	163.9	239.8		336.4 ok
c.100	4.0 J		3.0	1.9	2.1	J	4.0 J
c.101+090	244.6		267.6	211.3	236.9		237.4 ok
c.105	88.9		108.9	65.8	90.6		109.3 ok
c.107+147	25.2		13.1	0.0	0.0	U	0.0 U
c.110+077	216.3		236.1	153.0	197.7		184.0 ok
c.114	9.6		10.0	5.9	9.3		10.5 ok
c.116	283.9		345.2	199.1	309.4		381.3 ok
c.119	9.9		11.8	7.2	9.1		12.8 ok
c.124	4.1 J	U	0.0	0.0	1.6	J	0.0 U
c.126	0.7 J		0.4	0.3	0.8	J	0.7 ok
c.128	55.4		57.2	45.2	49.0		77.4 ok
c.129+126	4.8 J		4.9	3.2	4.5	J	3.1 J
c.130	24.3		24.9	19.5	21.9		32.2 ok
c.131	6.9 J		6.8	6.0	7.4	J	11.1 ok
c.132	32.3 J		54.5	43.0	52.2	J	81.5 J
c.134	0.0 U		0.0	0.0	0.0	U	0.0 U
c.135+144	11.9		12.3	10.9	14.9		10.3 ok
c.136	0.0 U		1.8	2.2	3.5	J	1.8 J
c.137	17.3		16.7	14.8	15.1		25.4 ok
c.136+160+183	363.3		390.6	335.4	333.3		622.1 ok
c.141+179	3.1 J		16.0	19.1	3.4	J	1.8 J
c.146	79.8		92.4	80.5	79.6		149.1 ok
c.149+123	130.0		115.9	113.1	106.3		152.2 ok
c.151	15.8		20.1	15.8	18.2		19.9 ok
c.153	540.9		439.2	412.8	375.4		660.4 ok
c.156	2.3 J		32.5	24.5	27.7		40.1 ok
c.158	25.7		25.2	23.8	21.5		41.8 ok
c.167	18.3		19.6	16.2	20.2		29.5 ok
c.169	0.8 J	U	0.5	0.4	0.1	J	0.3 U
c.170+190	59.6 J		60.3	53.3	46.0	J	134.0 J
c.171+202	25.2		26.3	25.5	26.5		57.1 ok
c.172	20.9		22.4	21.5	19.3		38.1 ok
c.173	1.1 J		1.2	0.8	1.7	J	6.9 J
c.174	26.5		24.3	29.3	22.0		48.6 ok
c.175	3.3 J		3.5	0.0	2.7	J	7.4 J
c.176	5.3 J		4.3	6.0	10.4		5.0 J
c.177	30.3		29.7	32.8	0.0	J	45.5 ok
c.178	14.1		15.6	13.5	17.2		30.2 ok
c.180	233.1		257.4	278.8	194.2		669.9 ok
c.183	54.5		57.2	61.8	47.9		129.9 ok
c.185	2.8 J		2.5	2.8	1.8	J	3.8 J
c.187+182	135.6		148.6	156.8	144.3		314.8 ok
c.189	3.3 J		3.7	2.8	2.8	J	6.6 J
c.191	3.4 J		3.5	3.6	2.1	J	6.6 J
c.193	10.6		12.7	12.1	9.7		23.2 ok
c.194	36.3		39.9	32.5	32.8		80.8 ok
c.195+208	17.9 J		23.9	15.6	22.3	J	44.3 J
c.197	2.9 J		2.9	2.6	2.6	J	5.6 J
c.198	3.1 J		3.2	2.6	2.7	J	5.2 J
c.199	36.2		41.6	32.9	39.7		69.0 ok
c.200	1.7 J		1.9	1.7	1.7	J	2.4 J
c.201+157	7.8 J		4.1	4.2	6.4	J	10.2 ok
c.203+196	47.8		52.1	43.8	45.7		102.8 ok
c.205	5.9 J		6.9	4.5	5.1	J	12.1 ok
c.206	23.8		24.5	15.1	25.9		50.2 ok
c.207	5.4 J		6.2	3.6	5.5	J	9.4 J
c.209	15.3 J		16.9	13.3	13.3	J	29.2 J
cong.sum	5426.3		5796.2	4319.4	4726.0		7096.0 ok
cong.sum_less85	5143.1		5796.2	4234.0	4694.7		7047.9 ok

0.1 J

0.1 J

# TECHNICAL PROCEDURES

## INTRODUCTION

The objective of this project was to prepare and analyze approximately 123 biota tissue samples to determine concentrations of polychlorinated biphenyl (PCB), and conduct related ancillary measurements. The PCB target analytes are listed in Attachment 2. Battelle analyzed fish and eggs that were collected between the spring of 1996 and the fall of 1996. The samples were shipped to Battelle in April, May, and June, 1997 and the Battelle laboratory component of this project began in early May, 1997.

## SAMPLE ANALYSIS

### SAMPLE RECEIPT, STORAGE, AND HOLDING TIMES

Hagler Bailly arranged for shipment of the frozen samples to Battelle. The samples were, upon receipt, logged into the laboratory and given unique Battelle IDs. The samples were stored frozen at, or below, -20°C until laboratory preparation could begin.

The tissue samples were stored frozen until they could be homogenized and composited. Homogenized and composited tissue samples were returned to frozen storage once they had been subsampled for extraction, or upon completion of the homogenization/compositing procedures, if extraction could not begin within one day. The sample holding times were 1 year from collection to extraction, as long as they were stored frozen until sample preparation begins. Sample extracts were to be analyzed within 40 days of extraction. Table 1 presents the 1 year holding time expiration dates. All samples were extracted by these dates and the extract holding times were also consistently met.

**Table 1. Fish and Egg Sample Holding Time Expiration Dates**

Sample Matrix	Holding Time Expiration Date
Walleye — whole body	July 29, 1997
Walleye — liver	July 29, 1997
Brown Trout — whole body	July 29, 1997
Brown Trout — fillet	October 11, 1997
Lake Trout — whole body	October 22, 1997
Lake Trout — fillet	August 12, 1997
Lake Trout — eggs	October 22, 1997
Tem — eggs	May 29, 1997

## PRELIMINARY SAMPLE COMPOSITING, SPLITTING, AND PREPARATION

The tissue was thawed and homogenized. A Hobart stainless steel grinder was used to homogenize the fillets and the whole body fish. This large-sample homogenate was collected in a stainless steel bowl, thoroughly mixed, and approximately 400 g removed for keep (the balance of the tissue homogenate was discarded). Each individual fish and fillet was homogenized and stored separately. A Tekmar Tissuemizer was used to further homogenize the fish fillet and whole body fish tissue that was used for laboratory analysis. The Tekmar Tissuemizer was also used to homogenize the livers and eggs. The homogenized sample was placed in a pre-cleaned glass jar, with Teflon lined cap, for subsequent storage. The final whole body walleye and brown trout samples were generated by compositing approximately 30 g ( $\pm 0.3$  g) aliquots of the homogenized tissue from several fish, and assigning this composite sample a new sample ID (in accordance with a compositing and sample ID scheme provided by Hagler Bailly). The number of samples prepared and analyzed are listed in Table 2.

Table 2. Number of Samples for Analysis

Sample Matrix	Base 106 PCB Congeners	Total PCB (as Aroclor)	Coplanar PCB Congeners
Walleye — whole body	31 <sup>a</sup>	0	5
Walleye — liver	0	17 <sup>b</sup>	0
Brown Trout — whole body	10 <sup>c</sup>	0	2
Brown Trout — fillet	0	14	0
Lake Trout — whole body	12 <sup>d</sup>	0	12
Lake Trout — fillet	0	15	0
Lake Trout — eggs	12	0	12
Tern — eggs	12	0	12
<b>Total:</b>	<b>77</b>	<b>46</b>	<b>43</b>

<sup>a</sup> The 31 walleye whole body samples were composited from 138 fish (3–6 fish/composite).

<sup>b</sup> The 17 liver samples were individual livers from 16 fish and one sample was the composite of livers from 4 fish.

<sup>c</sup> The 10 whole body brown trout samples were composited from 50 fish (4–6 fish/composite).

<sup>d</sup> The 12 lake trout whole body samples will each be of 1 fish (i.e., not composited).

## SAMPLE PREPARATION

The samples were analyzed in analytical batches of no more than 20 field samples per matrix type. The following eight (8) analytical batches were analyzed:

- 1 batch of walleye liver samples
- 2 batches of brown trout and lake trout fillet samples
- 1 batch of tern egg samples (6 forsters tern and 6 common tern eggs)
- 1 batch of lake trout egg samples
- 3 batches of combinations of walleye/brown trout/lake trout whole body samples

Additionally, there was one batch with a combination of walleye liver and trout fillet samples because several of these samples had relatively low recoveries the first time they were analyzed, and they were therefore re-analyzed in one batch.

The following quality control samples were processed along with the field samples (key quality control data quality objectives are listed in Attachment 3):

- 1 procedural blank (PB)
- 1 blank spike (BS)
- 1 certified reference material (CRM). The NRC material CARP-1 was used.
- 1 sample duplicate (DUP)

Additionally, equipment/rinse blank (EB) samples were generated during the homogenization process and instrument blanks (IB) were analyzed. The EB was a solvent (hexane) rinse of the sample homogenization equipment. One EB was prepared with each batch of samples. The IB was 1 mL of hexane that was fortified with internal standards and injected onto the GC/ECD. One IB was analyzed with each batch of samples, to determine if there was any instrument "background" signal. The EB and IB samples were quantified like the PB sample, and the average sample weight of the analytical batch was used to calculate concentrations.

### Tissue Extraction and Preparation

The tissue homogenate sample was thoroughly mixed and approximately 3–10 g was removed for the extraction (Table 3). The amount of tissue used for the extraction, and the eventual pre-injection volume (PIV) the sample was adjusted to, depended on the expected PCB congener concentrations (as communicated to Battelle by Hagler Bailly during the planning phase of this project). The sample was fortified with surrogate internal standards [SISs: PCB congeners Cl<sub>3</sub>(36) and Cl<sub>3</sub>(112)] to monitor procedural efficiency. Sodium sulfate was added to dry the sample and aid in the maceration, and the sample was serially extracted three times in a Teflon jar using hexane as the extraction solvent and a Tekmar Tissuemizer. The combined extract concentrated using a Kuderna-Danish apparatus and gentle nitrogen gas evaporation on an N-Evap.

**Table 3. Target Weight and PIVs**

Sample Matrix	Approximate Sample Weight Extracted (g)	Approximate Pre-Injection Volume (mL) <sup>a</sup>
Walleye — whole body	5	2
Walleye — liver	3	10
Brown Trout — whole body	5	4
Brown Trout — fillet	10	2
Lake Trout — whole body	5	2
Lake Trout — fillet	10	2
Lake Trout — eggs	5	4
Tem — eggs	5	10

<sup>a</sup> The PIV for the base congener analysis was half of this if the sample was split for coplanar congener analysis.

The extract was next purified using a chromatography column packed with 20-g, 2% deactivated F-20 alumina (a 40-g alumina column was used for the egg samples). The column eluant was concentrated using Kuderna-Danish technique and further purification was obtained by serially treating the extract with sulfuric acid until there was no visible reaction.

The alumina and sulfuric acid purified sample was concentrated using Kuderna-Danish and nitrogen evaporation techniques and adjusted to the desired PIV. If coplanar PCB analysis was to be performed, the final extract was split 50:50, with one half being submitted for coplanar PCB fractionation (see Coplanar PCB Congener Determination below) and the other half fortified with recovery internal standards [RIS: PCB congeners Cl<sub>3</sub>(34), Cl<sub>3</sub>(39), and Cl<sub>6</sub>(166)] and submitted for instrumental analyses.

#### **Ancillary Measurements**

Moisture and lipid content was determined following standard gravimetric protocols. In summary, the lipid content was determined as the "hexane extractable matter" by subsampling 10 mL of the approximately 200 mL combined sample extract, allowing it to dry and weighing the material twice at least 1 hour apart to ensure complete solvent evaporation. The volume of the sample extract, from which the subsample was removed for the lipid determination, was accurately measured by marking the volume level on the outside of the glassware prior to removing the subsample for the lipid measurement. Once the balance of the extract had been transferred for concentration, the original extract volume was determined by pouring water into the glassware to the marked level and measuring the volume using a graduated cylinder.

In addition to the hexane extractable lipid determination, which was performed on all samples, three whole body trout samples were extracted separately using dichloromethane (DCM) for determination of the DCM extractable lipid content. This was performed to obtain data to compare the lipid data generated with the standard hexane extraction with that obtained using DCM.

The moisture content was determined by placing approximately 5 g of wet tissue material in a tarred weighing pan, which was then dried at least 24 hours in a drying oven at 105°C. The dry material was then removed from the oven and allowed to come to room temperature before it was again weighed. The weighing was repeated at least 6 hours later to ensure complete dryness.

#### **Coplanar PCB Congener Determination**

A sub-set of the samples analyzed for ortho substituted PCB congeners ("standard" congeners) were also analyzed for coplanar (non-ortho substituted) PCB congeners. A total of 26 samples were processed and analyzed for coplanar PCB congeners.

The final purified extract prepared for standard PCB congener analysis was split 50:50 prior to the addition of the RIS, as described above. The coplanar PCB congener analysis was performed on one of the two splits, after isolating the coplanar congeners in accordance with Draft EPA Method 1668, *Toxic PCBs by HRGC/HRMS*:

Approximately 25 ng of the coplanar PCB congener SIS [Cl<sub>4</sub>(77)-deuterated] was added to the coplanar extract split to monitor the efficiency of the column separation and coplanar PCB congener isolation. A 9-mm glass column was packed with 3.6 g of a 50:50 mixture of Carbopack C: Celite 545 that had been activated at 130°C for a minimum of 6 hours; the column was packed in hexane. The sample extract was loaded onto the column, rinsing the sample vial with approximately 1 mL of hexane, which was added to the column. The solvent level was brought to the top of the column and the column eluted as follows:

- 25 mL of hexane was added, eluted, and collected as the F1 (standard congeners).
- 15 mL of methanol was added, eluted, and collected as the F2 (residual polar/lipid matrix components).
- 15 mL of toluene was added, eluted, and collected as the F3 — the coplanar PCB congeners elute in this fraction.

The F3 fraction (coplanar PCB congener) was concentrated to approximately 200–250 µL using nitrogen evaporation techniques, fortified with the RIS compounds, and submitted for GC/ECD analysis.

## INSTRUMENTAL ANALYSIS

### GC/ECD Analysis — PCB Congener Analysis

The analysis of the target PCB congener compounds (Attachment 2) was performed by high-performance capillary gas chromatography with electron capture detection (GC/ECD) using a Hewlett-Packard 6890 or 5890-II gas chromatograph fitted with dual  $^{63}\text{Ni}$ -electron capture detectors. The GC/ECD analysis was performed using a 60-m, 0.25-mm inner diameter, 0.25- $\mu\text{m}$  film thickness, DB-5 fused silica capillary column (J&W Scientific, Inc.). A 1  $\mu\text{L}$  sample extract was injected onto the instrument. The injected sample was also split to a second column (60-m, 0.25-mm inner diameter, 0.25- $\mu\text{m}$  film thickness, DB-1701 column) and ECD, for simultaneous acquisition of second column GC/ECD data. The second column were acquired in case these data would be needed for review at a later time, but the analyses on the DB-1701 will not be calibrated and the data were not reduced for this project (the DB-1701 runs were, however, checked to ensure that the data were acquired).

The GC was equipped with an electronic pressure controlled (EPC) inlet for optimum sensitivity and reproducibility. Additionally, hydrogen was used as the carrier gas, and the temperature program was optimized to separate the 106 target PCB congeners. The following GC temperature program was used:

- Initial temperature 60 °C
- Initial time 1 minute
- Ramp Rate 10 °C/minute to 140 °C; 1 °C/minute to 220 °C; 5 °C/minute to 290 °C
- Final temperature 290 °C; 10 minutes

The GC/ECD system was calibrated with a multilevel calibration, with a minimum of 4 calibration points (5 points were typically be used). The analyte concentrations range from about 0.005 to 0.12  $\text{ng}/\mu\text{L}$  in the calibration solutions (the concentrations of some congeners was higher, because of their lower ECD response), and the internal standard concentrations were approximately 0.05 to 0.06  $\text{ng}/\mu\text{L}$  in all calibration levels. The calibration solutions were prepared with all 106 target congeners and the internal standards. For the coeluting sets of congeners (see Attachment 2), only the primary congener (the congeners listed first in Attachment 2) was used in the calibration solutions.

Each target analyte was fitted to a quadratic equation to best represent the response of the ECD. The validity of the initial calibration was monitored with a continuing calibration check analysis ( a midlevel calibration standard) at least every 10 samples. Analytes concentrations were by the method of internal standards using the RIS (i.e., the internal standard added at the end of the sample processing regime) as the quantification internal standard.

Samples with target PCB congeners response above the high standard were diluted and re-analyzed. If more than 10 of the PCB congeners had a response greater than the high calibration standard, then the analytical data from only the diluted run were reduced and reported. However, if the dilution and re-analysis was performed because 10 or fewer PCB congeners had a response above the high calibration standard, then the data for all congeners were reported from the first run, and the re-analysis was only used to generate data for the congeners that were initially above the high standard (and the "E" and "D" qualifiers applied to the data, as described in Attachment 4).

Quantification of individual components was performed by the method of internal standards using the RIS compounds C13(39) and C16(166) as the quantification internal standard [C13(39) was used for all congeners eluting before the SIS C15(112), and C16(166) was used for the congeners eluting after this SIS]. Surrogate compound recoveries were determined for the SIS C13(36) versus the RIS C13(39), and for the SIS C15(112) versus the RIS C16(166). Target analyte concentrations were reported on a wet weight basis, and the moisture and lipid content were reported along with the PCB analytical data.

Additionally, the total PCB was estimated as the sum of the 106 congener concentrations on the spreadsheet summary tables. The sum of all congeners without congener #85 was also calculated because there was a significant interference observed with this congener and this likely biased the total PCB data when this congener was included.

#### **GC/ECD Analysis — Total PCB Analysis (as Aroclor Equivalent)**

The total PCB concentrations in the walleye liver and brown trout and lake trout fillet was determined by the Aroclor equivalent method; no individual PCB congener data were generated for these samples. The sample extraction and preparation was the same as for the PCB congener analysis, and the instrumental analysis was also as described above except for the calibration standards that was used.

The initial calibration verification was performed with a multilevel calibration containing a mixture of Aroclors 1016 and 1260 (with a concentration range of approximately 0.25 to 5  $\mu\text{g/mL}$  per Aroclor). Single-point calibration standards were analyzed for the other target Aroclor formulations (Attachment 2). Additionally, a 50:50 mixture of Aroclors 1248:1254 was analyzed as a single-point calibration standard. The validity of the calibration was checked with a mid level calibration mixture of 1016 and 1260 (with a concentration of approximately 2  $\mu\text{g/mL}$ ) no less frequently than every 10 samples. The multilevel calibration would be used to quantify the samples that most closely resemble 1016 and 1260, and the appropriate single-point calibrations was used for the other Aroclor formulations.

Total PCB was determined as the most predominant Aroclor formulation (i.e., the analysts reviewed the chromatogram and determined which single Aroclor the PCB composition in the sample most closely resembles, and quantified the sample as the equivalent of that Aroclor). Because the Aroclor composition relatively closely resembled a 50:50 mixture of Aroclors 1248 and 1254 (ranged from about 40:60 to 60:40) in the walleye liver samples, the standard with a 50:50 of these Aroclors was used to quantify those samples, and the results were reported as "1248.1254". The PCB pattern in the trout filets most closely resembled Aroclor 1254, and this formulation was used to quantify the filets.

The RIS C16(166) was used as the quantification internal standard for Aroclors 1248, 1254, and 1260, and the RIS C13(39) was used for Aroclors 1221, 1016, 1232, and 1242. The RIS C13(39) was also used to determine the recovery of the SIS C13(36) and the RIS C16(166) was used to determine the recovery of the SIS C15(112).

#### **GC/ECD Analysis — Coplanar PCB Congener Analysis**

Samples selected for coplanar PCB congener analysis were processed for the isolation of these congeners, and separately submitted for GC/ECD analysis. The GC analytical conditions was the same as for the analysis of standard PCB congeners. The same calibration and quantification approach was also used for the coplanar congeners. Sample quantification will be performed versus the RIS C16(166) for all coplanar congeners except congener #37; the RIS C13(39) was used to quantify congener #37. The recovery of the column fractionation internal standard C14(77)-*deuterated* was determined versus the RIS C16(166). The recovery of C14(77)-*deuterated* was an indicator of the sample processing efficiency after the sample was split for coplanar PCB congener processing. The efficiency of the rest of the sample processing was indicated by the recoveries of the standard SISs [C13(36) and C15(112)], which were reported with the standard PCB congener analysis data for each sample.

#### **GC/MS Confirmatory Analysis**

The quantity of the standard PCB congeners was confirmed using quadrupole gas chromatography with mass spectrometric detection using a Hewlett-Packard Model 5972 MSD. All field samples that were analyzed for the base congeners by GC/ECD (77 samples) were also analyzed by GC/MS. However, the GC/MS data were only reduced and reported for 26 of the 77 samples; 4 tern egg, 4 lake trout egg, 10

walleye whole body, 4 brown trout whole body, and 4 lake trout whole body samples. The 26 samples were selected using criteria developed by Hagler Bailly (species, tissue type, location of capture, and PCB concentration determined in the GC/ECD analysis). GC/MS confirmation was not performed on the coplanar PCB congener samples or the samples that were analyzed for total PCB as Aroclor equivalent. The GC/MS analysis was performed on the field samples and the PB samples — the QC data, including surrogate compound recoveries, were generated from the GC/ECD analyses.

The gas chromatograph was fitted with the same chromatography column and operated with the same oven temperature profile as that used for the primary GC/ECD analysis. However, helium was used as the carrier gas instead of hydrogen. This ensure that the peaks tentatively identified by GC/ECD had comparable chromatographic properties in the GC/MS analysis. However, because helium was used as the carrier gas instead of hydrogen, congeners #153 and #132, which were resolved on the GC/ECD, could not be resolved in the GC/MS analysis of the whole body fish samples (they were resolved during the analysis of the egg samples).

The mass spectrometer was operated in the selected ion monitoring mode (SIM) to provide the necessary sensitivity and selectivity. Each target congener was monitored using two ions — a primary ion for quantitation and a secondary ion, for structural identification and confirmation. Identifications was based on chromatographic retention time and primary/secondary ion ratio criteria (i.e., identification of the peak as a PCB congener, the level of chlorination of that PCB congener, and the known retention time characteristics of each congener from prior detailed GC/ECD retention time characterization/mapping).

The GC/MS analytical system was tuned with perfluorotributylamine (PFTBA), and calibrated with a multilevel calibration. A minimum of 4 calibration levels (but typically 5 points), was used with the analyte concentrations in a range of approximately 0.02 to 0.8 ng/ $\mu$ L. The calibration solutions contained all 106 target base congeners and the internal standards. The GC/MS analytes were quantified versus the RIS C13(34).

The GC/MS confirmatory analysis was performed like a standard quantitative analysis, with the GC/MS data being reported just like the GC/ECD data. There was no quantitative comparison of the GC/ECD and GC/MS analytical results.

## **MDL STUDY**

A method detection limit (MDL) study was performed as part of this project using "clean" (hatchery raised) trout fillet provided by Hagler Bailly. The MDL study was performed in accordance with the EPA protocol set forth in 40 CFR 136 Appendix B Method Detection Limit (MDL) Determination.

The MDL study involved fortifying eight replicate tissue homogenate sub-samples with the 106 target base PCB congeners at a concentration of approximately 3 to 5 times the expected MDL, and processing and analyzing them using the procedure that was used for the project field samples. Additionally, two non-spiked sub-samples were analyzed to determine the background PCB levels in the tissue, and a procedural blank analysis will also be included. The PCB congener concentrations were determined by GC/ECD, and the summary statistics performed to calculate the MDL for each PCB congener.

## QUALITY ASSURANCE / QUALITY CONTROL

### QUALITY ASSURANCE

The Quality Assurance Unit (QAU) at Battelle remains independent of all laboratory project activities. The QAU monitored Battelle's components of the project according to existing Battelle SOPs to ensure the accuracy, integrity, and completeness of the data. Additionally, the QAU monitored the project activities to ensure consistency with the applicable requirements described in the Quality Assurance Project Plan (QAPP) that was developed for this project. The QAU scope included system inspections, data audits, and reviews of documents and deliverables.

### QUALITY CONTROL

Project staff were responsible for ensuring that sample tracking, sample preparation, and analytical instrument operation all met the quality control criteria detailed in the applicable analytical SOPs. The type and frequency of analysis of quality control samples for the analyses are specified in Attachment 3.

The data quality objectives (DQO) for the analyses are outlined in Attachment 3. Analytical results that did not meet the listed DQOs were submitted to and/or reviewed with the Battelle Project Manager for assessment of the potential impact of the results. Affected samples were reanalyzed at the Project Manager's discretion (e.g., a set of samples were re-extracted and re-analyzed for total PCB as Aroclor determination because surrogate recoveries fell below the DQO). Quality control sample data that were accepted outside these criteria are indicated with the appropriate data qualifier. A set of data qualifiers were applied to the final summary spreadsheet data, as indicated in Attachment 4 (e.g., quality control sample data quality objective exceedances will be qualified with a "&" on the summary spreadsheet tables). Target analyte concentrations were reported if the analyst could confidently perform the identification and determination (i.e., uncensored data were reported).

**Attachment 2. Target PCB Analytes**

<b>Base PCB Congener Set <sup>a</sup></b>				
1	42/37	89	136	183
3	43	91	137	185
4/10	44	92	138/160/163	187/182
6	45	95	141/179	189
7/9	46	97	146	191
8/5	47/75	99	149/123	193
12/13	48	100	151	194
16/32	49	101/90	153	195/208
17/15	51	105	156	197
18	52	107/147	158	198
19	53	110/77	167	199
21	56/60	114 <sup>b</sup>	169	200
22	59	118	170/190	201/157
24/27	63	119	171/202	203/196
25	66	124	172	205
26	70/76	128	173	206
28	74	129/126	174	207
29	82	130	175	209
31	83	131	176	
33/20	84	132	177	
40	85 <sup>b</sup>	134	178	
41/64/71	87/115/81	135/144	180	

<sup>a</sup> All congeners numbers are listed using the IUPAC nomenclature.

Coeluting congeners are listed in order of abundance in Aroclors 1242/1248/1254 (most abundant listed first). The most abundant single congener will be used to calibrate the instrument for the coeluting congener sets.

<sup>b</sup> The pesticide 4,4-DDD coelutes with congener 114 and the pesticide 4,4-DDE coelutes with congener 85.

Attachment 2 (cont.). Target PCB Analytes

<b>Coplanar PCB Congener Set</b>
37
77
81
126
169
<b>Aroclor Formulations</b>
Aroclor 1016
Aroclor 1221
Aroclor 1232
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 1260

**Attachment 3. Data Quality Objectives – PCB Analysis**

QC Sample	Frequency	Data Quality Objectives	Corrective Action
Procedural Blank (PB)	1 per analytical batch <sup>a</sup>	< RL, or associated samples >10 × blank concentration	Reanalyze associated samples, or justify.
Equipment Blank (EB)	1 per analytical batch	< RL, or associated samples >10 × blank concentration	Qualify data and/or describe in narrative with data reporting, or justify.
Instrument Blank (IB)	1 per analytical batch	< RL, or associated samples >10 × blank concentration	Reanalyze associated samples, or justify.
Blank Spike (BS)	1 per analytical batch <sup>a</sup>	90% of congeners to meet the following: 50–125% recovery for tri- through decachlorobiphenyls and Aroclors. 30–125% recovery for mono- and dichlorobiphenyls.	Reanalyze associated samples, or justify.
Certified Reference Material (CRM) (CARP-1)	1 per analytical batch	PD <±35% between measured and certified or consensus value for 90% of analytes; PD <±50% for all analytes. Average of PD (absolute values) <25%. Objectives apply to analytes with a certified or consensus concentration >5 × RL.	Reanalyze associated samples, or justify.
Sample Duplicate (DUP)	1 pair per analytical batch <sup>a</sup>	RPD <50% for duplicates with analyte concentrations >5 × RL. Difference <2 × RL for duplicates with analyte concentration <5 × RL.	Reanalyze associated samples, or justify.
Surrogate Compounds	Every field and QC sample	50–125% recovery	Reanalyze associated samples, or justify.
Initial Instrument Calibration (GC/ECD and GC/MS)	At initiation of analytical sequence.  A minimum of 4-point calibration.	GC/ECD: Correlation coef. r >0.995 for 90% of analytes; r >0.99 for all analytes. (r >0.995 = r <sup>2</sup> >0.99)  GC/MS: < 25% RSD in RRFs for 90% of analytes; <35% RSD for all analytes.	Recalibrate and reanalyze associated samples

<sup>a</sup> Analytical Batch: Sample set of no more than 20 field samples of the same sample matrix.

Attachment 3 (cont.). Data Quality Objectives – PCB Analysis

<p>Continuing Instrument Calibration Check  (GC/ECD and GC/MS)</p>	<p>No less frequently than every 10 samples</p>	<p>GC/ECD: determined concentration <math>&lt;\pm 25\%</math> PD vs true concentration for 75% of analytes. <math>&lt;\pm 35\%</math> PD for 90% of analytes; <math>\pm 50\%</math> PD for all analytes. <math>&lt;\pm 15\%</math> PD on average for all analytes.</p> <p>GC/MS: <math>&lt;\pm 25\%</math> PD for RRFs versus initial calibration for 75% of analytes; <math>&lt;\pm 35\%</math> PD for 90% of analytes; <math>\pm 50\%</math> PD for all analytes. <math>&lt;\pm 15\%</math> PD on average for all analytes.</p>	<p>Recalibrate and reanalyze associated samples (i.e., samples not bracketed by a passing calibration), or justify.</p>
<p>% Lipid determination</p>	<p>Replicate weighing of each sample.  Sample duplicate — 1 per analytical batch <sup>a</sup></p>	<p><math>&lt;10\%</math> difference in two weighings  RPD <math>&lt; 20\%</math></p>	<p>Re-dry and re-weigh  Qualify data.</p>
<p>% Moisture determination</p>	<p>Replicate weighing of each sample.  Sample duplicate — 1 per analytical batch <sup>a</sup></p>	<p><math>&lt;10\%</math> difference in two weighings  RPD <math>&lt; 20\%</math></p>	<p>Re-dry and re-weigh.  Re-determine moisture content for associated samples, or justify.</p>

#### Attachment 4. Data Qualifiers

Data Qualifier	Purpose
&	QC value outside the accuracy or precision criteria goal (SRM, BS recovery, surrogate recovery, %RPD in DUP analysis).
E	Value for analysis of compound with response above the calibration range. Sample was diluted and reanalyzed for this analyte, and the data from the diluted sample analysis are reported separately elsewhere.
D	Value for diluted analysis of compound with an original (undiluted) response above the high calibration range.
B	Analyte detected at a level above the reporting limit in the procedural blank (procedural blank value is qualified).
U	Not detected. An entry of "0" is put in the value field.
J	Estimated value. Analyte detected below the sample-specific reporting limit.
ME	Significant matrix interference — estimated value reported.
MI	Significant matrix interference — value could not be determined or estimated.
JX	Estimated value, see narrative <sup>b</sup> .
X, Y, Z	Defined in case narrative.

<sup>a</sup> Data qualifiers that will be applied to the summary spreadsheet. Qualifying uses RLs and analyst compound identification; calculated MDLs are not used when applying the "U" or "J" qualifiers, and there is currently no qualifier for values between the MDL and the RL, or that use the MDL in any way.

<sup>b</sup> The JX qualifier was specifically created to qualify the congener #85 data, which in the field samples was uncharacteristically large, likely due to interference from the pesticide p,p'-DDE. This is described in more detail in a narrative in the letter data report.

## Attachment 5

## REPORTING LIMITS - Extended PCB Congener Set

<i>Batch ID</i>	97-126	97-129	97-190, 97-191, 97-192	97-191, 97-192	97-192
<i>Matrix</i>	<i>Tem eggs</i>	<i>L. Trout Eggs</i>	<i>Walleye Whole</i>	<i>B.Trout Whole</i>	<i>L.Trout Whole</i>
Pre-injection Volume (uL)	5000	2000	1000	2000	1000
Lipid Analysis Split Factor	1.00	1.00	1.00	1.00	1.00
Coplanar Analysis Split Factor	2.00	2.00	1.00	1.00	1.00
Sample Dilution Factor	1.00	1.00	1.00	1.00	1.00
Sample Wet Weight (g)	5.00	5.00	5.00	5.00	10.00
Reporting Unit	ng/g, wet weight	ng/g, wet weight	ng/g, wet weight	ng/g, wet weight	ng/g, wet weight
<b>Analyte</b>					
PCB1	25.6	10.2	2.6	5.1	1.3
PCB3	50.2	20.1	5.0	10.0	2.5
PCB4	25.8	10.3	2.6	5.2	1.3
PCB7	16.0	6.4	1.6	3.2	0.8
PCB6	16.0	6.4	1.6	3.2	0.8
PCB8	16.9	6.7	1.7	3.4	0.8
PCB19	9.0	3.6	0.9	1.8	0.5
PCB12	16.0	6.4	1.6	3.2	0.8
PCB18	8.4	3.4	0.8	1.7	0.4
PCB17	7.2	2.9	0.7	1.4	0.4
PCB24	9.0	3.6	0.9	1.8	0.5
PCB16	9.0	3.6	0.9	1.8	0.5
PCB29	9.0	3.6	0.9	1.8	0.5
PCB26	9.0	3.6	0.9	1.8	0.5
PCB25	9.0	3.6	0.9	1.8	0.5
PCB31	9.0	3.6	0.9	1.8	0.5
PCB28	8.4	3.4	0.8	1.7	0.4
PCB21	9.0	3.6	0.9	1.8	0.5
PCB33	9.0	3.6	0.9	1.8	0.5
PCB53	9.6	3.8	1.0	1.9	0.5
PCB51	6.2	2.5	0.6	1.2	0.3
PCB22	9.0	3.6	0.9	1.8	0.5
PCB45	7.8	3.1	0.8	1.6	0.4
PCB46	9.8	3.8	1.0	1.9	0.5
PCB52	8.4	3.4	0.8	1.7	0.4
PCB43	9.0	3.6	0.9	1.8	0.5
PCB49	9.6	3.8	1.0	1.9	0.5
PCB47	9.8	3.8	1.0	1.9	0.5
PCB48	9.6	3.8	1.0	1.9	0.5
PCB44	8.4	3.4	0.8	1.7	0.4
PCB59	9.6	3.8	1.0	1.9	0.5
PCB42	9.0	3.6	0.9	1.8	0.5
PCB41	7.4	3.0	0.7	1.5	0.4
PCB40	9.0	3.6	0.9	1.8	0.5
PCB100	9.6	3.8	1.0	1.9	0.5
PCB63	9.6	3.8	1.0	1.9	0.5
PCB74	9.6	3.8	1.0	1.9	0.5
PCB70	9.6	3.8	1.0	1.9	0.5
PCB66	8.4	3.4	0.8	1.7	0.4
PCB95	9.6	3.8	1.0	1.9	0.5
PCB91	7.4	3.0	0.7	1.5	0.4
PCB56	7.6	3.0	0.6	1.5	0.4
PCB92	6.8	2.7	0.7	1.4	0.3
PCB84	7.6	3.0	0.8	1.5	0.4
PCB89	9.6	3.8	1.0	1.9	0.5
PCB101	8.4	3.4	0.8	1.7	0.4
PCB99	9.6	3.8	1.0	1.9	0.5
PCB119	9.6	3.8	1.0	1.9	0.5
PCB83	9.6	3.8	1.0	1.9	0.5

REPORTING LIMITS - Extended PCB Congener Set

Batch ID Matrix	97-126 Tern eggs	97-129 L. Trout Eggs	97-190, 97-191, 97-192 Walleye Whole	97-191, 97-192 B. Trout Whole	97-192 L. Trout Whole
PCB97	9.6	3.8	1.0	1.9	0.5
PCB67	9.6	3.8	1.0	1.9	0.5
PCB85	7.4	3.0	0.7	1.5	0.4
PCB136	9.6	3.8	1.0	1.9	0.5
PCB110	9.6	3.8	1.0	1.9	0.5
PCB82	9.6	3.8	1.0	1.9	0.5
PCB151	9.6	3.8	1.0	1.9	0.5
PCB135	7.0	2.8	0.7	1.4	0.4
PCB124	9.6	3.8	1.0	1.9	0.5
PCB107	9.6	3.8	1.0	1.9	0.5
PCB149	9.6	3.8	1.0	1.9	0.5
PCB118	8.4	3.4	0.8	1.7	0.4
PCB134	9.6	3.8	1.0	1.9	0.5
PCB114	9.6	3.8	1.0	1.9	0.5
PCB131	8.6	3.4	0.8	1.7	0.4
PCB146	7.0	2.8	0.7	1.4	0.4
PCB153	8.4	3.4	0.8	1.7	0.4
PCB132	9.6	3.8	1.0	1.9	0.5
PCB105	8.4	3.4	0.8	1.7	0.4
PCB141	9.6	3.8	1.0	1.9	0.5
PCB137	9.6	3.8	1.0	1.9	0.5
PCB176	7.8	3.1	0.8	1.6	0.4
PCB130	7.8	3.0	0.8	1.5	0.4
PCB138	8.4	3.4	0.8	1.7	0.4
PCB158	9.8	3.8	1.0	1.9	0.5
PCB129	9.6	3.8	1.0	1.9	0.5
PCB178	8.8	2.7	0.7	1.4	0.3
PCB175	9.6	3.8	1.0	1.9	0.5
PCB187	8.4	3.4	0.8	1.7	0.4
PCB183	8.4	3.4	0.8	1.7	0.4
PCB128	8.4	3.4	0.8	1.7	0.4
PCB167	9.6	3.8	1.0	1.9	0.5
PCB185	9.6	3.8	1.0	1.9	0.5
PCB174	8.0	2.4	0.6	1.2	0.3
PCB177	5.8	2.3	0.6	1.2	0.3
PCB171	9.6	3.8	1.0	1.9	0.5
PCB156	9.6	3.8	1.0	1.9	0.5
PCB173	9.6	3.8	1.0	1.9	0.5
PCB201	9.6	3.8	1.0	1.9	0.5
PCB172	9.6	3.8	1.0	1.9	0.5
PCB197	9.6	3.8	1.0	1.9	0.5
PCB180	8.4	3.4	0.8	1.7	0.4
PCB183	9.8	3.8	1.0	1.9	0.5
PCB191	9.6	3.8	1.0	1.9	0.5
PCB200	9.8	3.8	1.0	1.9	0.5
PCB169	9.6	3.8	1.0	1.9	0.5
PCB170	8.4	3.4	0.8	1.7	0.4
PCB198	9.6	3.8	1.0	1.9	0.5
PCB199	8.0	3.2	0.8	1.6	0.4
PCB203	7.6	3.0	0.8	1.5	0.4
PCB189	9.6	3.8	1.0	1.9	0.5
PCB195	8.4	3.4	0.8	1.7	0.4
PCB207	7.8	3.1	0.8	1.6	0.4
PCB194	9.8	3.8	1.0	1.9	0.5
PCB205	9.6	3.8	1.0	1.9	0.5
PCB206	6.4	2.6	0.6	1.3	0.3
PCB209	6.4	2.6	0.6	1.3	0.3

Attachment 5 (cont.)

REPORTING LIMITS - Coplanar Congeners

<i>Batch ID</i>	97-126	97-129	97-192	97-192	97-192
<i>Matrix</i>	<i>Tern eggs</i>	<i>L. Trout Eggs</i>	<i>Walleye Whole</i>	<i>B. Trout Whole</i>	<i>L Trout Whole</i>
Pre-Injection Volume (uL)	125	125	200	200	200
Lipid Analysis Split Factor	1.00	1.00	1.00	1.00	1.00
Coplanar Analysis Split Factor	2.00	2.00	2.00	2.00	2.00
Sample Dilution Factor	1.00	1.00	1.00	1.00	1.00
Sample Wet Weight (g)	5.00	5.00	5.00	5.00	10.00
Reporting Unit	ng/g, wet weight		ng/g, wet weight	ng/g, wet weight	ng/g, wet weight
<i>Analyte</i>					
PCB37	0.24	0.24	0.38	0.38	0.19
PCB81	0.24	0.24	0.38	0.38	0.19
PCB77	0.24	0.24	0.38	0.38	0.19
PCB126	0.24	0.24	0.38	0.38	0.19
PCB169	0.25	0.25	0.40	0.40	0.20

Attachment 5 (cont.)

REPORTING LIMITS - Aroclors

<i>Batch ID</i>	97-124	97-127	97-128
<i>Matrix</i>	<i>Walleye Liver</i>	<i>B. Trout Fillet</i>	<i>L. Trout Fillet</i>
Pre-Injection Volume (uL)	10000	1000	1000
Lipid Analysis Split Factor	1.00	1.00	1.00
Coplanar Analysis Split Factor	1.00	1.00	1.00
Sample Dilution Factor	1.00	1.00	1.00
Sample Wet Weight (g)	3.00	10.00	10.00
Reporting Unit	ng/g, wet weight	ng/g, wet weight	ng/g, wet weight
<b>Analyte</b>			
Aroclor1016	816.7	24.5	24.5
Aroclor1221	816.7	24.5	24.5
Aroclor1232	816.7	24.5	24.5
Aroclor1242	816.7	24.5	24.5
Aroclor1248	816.7	24.5	24.5
Aroclor1248,1254	816.7	24.5	24.5
Aroclor1254	816.7	24.5	24.5
Aroclor1260	816.7	24.5	24.5

## Attachment 5 (cont.)

**Reporting Limit Calculation (and Spreadsheet Header Information).** Sample-specific RLs are calculated directly in the Excel summary tables for application of "J" qualifiers. The reporting limits listed in the table in Attachment 5 are based on the most common PIVs for the type, lipid analysis split factor of 1.00, a dilution factor of 1.00 (no dilution), and the targeted sample weight for the sample type. Sample-specific RLs, that were actually used to qualify the data, can be calculated by using the actual factors, weights etc. listed in the spreadsheet table heading for each individual sample.

The RLs are calculated as follows:

$$RL = \text{STD CONC} \times \text{PIV} \times \text{Lipid}_{\text{SF}} \times \text{Coplanar}_{\text{SF}} \times \text{Sample}_{\text{DF}} \times 1 / \text{Sample}_{\text{WT}}$$

RL = Reporting limit (ng/g, wet weight)

STD CONC = PCB concentration in low-level calibration standard (ng/ $\mu$ L)

PIV = Pre-injection volume ( $\mu$ L). The PIV listed in the Excel data table header is used for calculating RLs, and is determined slightly differently than what is typically thought of as a PIV. Pre-injection volume in this case is the final adjusted extract volume that contains the sample for the subject analysis. This is not necessarily the same as the volume of extract that is spiked with RIS or the volume of extract placed on the GC for analysis. In the case of most analyses it is the volume the sample is adjusted to prior to analysis (and not what is removed to place on the instrument), while in the case of diluted samples it is the volume the sample is adjusted to during the dilution (and, again, not what is removed to place on the instrument). In the case of the livers, the PIV entered here is 10,000  $\mu$ L because this is the adjusted volume of the entire final sample extract.

Lipid<sub>SF</sub> = Lipid analysis subsampling factor. Factor that corrects for the amount removed in the lipid analysis.  $(\text{total extract volume before lipid analysis}) \times (1 / (\text{total extract volume before lipid analysis} - \text{extract volume removed for lipid analysis}))$

Coplanar<sub>SF</sub> = Coplanar analysis split factor. Factor that corrects for any splitting of the extract for coplanar PCB analysis.  $(\text{total extract volume before split} / \text{extract volume removed for the subject analysis})$

Sample<sub>DF</sub> = Sample dilution factor. Factor that corrects for any subsampling of the extract for dilution purposes (i.e., when samples were diluted and re-analyzed). This is only a factor when a portion of the extract is removed, and subsequently spiked with additional RIS. The amount of solvent added to perform the dilution is not a factor in the calculation. Additionally, this is *not* a factor if only the PIV is increased to bring the analyte response within the calibration range in a re-analysis.  $(\text{total extract volume before subsampling} / \text{extract volume removed for the subject dilution and re-analysis})$

Sample<sub>WT</sub> = Sample weight (g, wet weight). Weight of the sample amount that was extracted.

Attachment 6

LIPID METHOD COMPARISON - Hexane vs Dichloromethane

Client Reporting ID	Matrix	Battelle ID	Analytical Batch	Lipid Content (% wet weight)		PD
				Hexane as Solvent	DCM as Solvent	
BTEG01CP	Brown trout whole body	VD38	97-191	11.42	12.09	5.9
BTEG02CP	Brown trout whole body	VD39	97-191	8.67	12.00	38.4
BTEG04CP	Brown trout whole body	VD40	97-191	11.20	15.76	40.7
					Average:	28.3

PD: percent difference; DCM relative to hexane.

Attachment 7

DUPLICATE MOISTURE CONTENT DETERMINATION

Client Reporting ID	Matrix	Battelle ID	Analytical Batch	% Moisture		RPD
				1:st Determination	2:nd Determination	
WEWG02LV	Walleye liver	VA44	97-124	61.94	41.83	38.8
BTEG01FC-1	Brown trout fillet	Z5981	97-127	77.74	77.65	0.1
LTLM01FC-1	Lake trout fillet	Z5874	97-128	67.43	61.81	8.7
LTIR08FC-1	Lake trout fillet	Z5858	97-181	67.95	61.21	10.4
TEKIB06	Tern egg	Z5797	97-126	78.44	57.51	30.8
EGLMF01FC-1	Lake trout egg	Z5958	97-129	66.78	69.41	3.9
WEFR01CP	Walleye whole body	VC53	97-190	61.04	62.46	2.3
WEEG04CP	Walleye whole body	VC73	97-191	58.70	67.42	13.8
WEFR07CP	Walleye whole body	VC59	97-192	65.38	62.22	5.0
Average:						12.6

## Attachment 8

### QUANTIFICATION OF SAMPLES -- PCB by GC/ECD

Samples are quantified using the method of internal standards. The quantification internal standards are the recovery internal standards (RIS) (i.e., the internal standards added to the sample immediately prior to instrumental analysis). The concentration of target analytes is determined using the following regression equation if a linear regression calibration is used:

$$C_s = [(A_s/A_i) - b] * (Amt_i/m) * (1/W)$$

Which is based on the linear regression equation:

$$Y = mX + b \text{ which is equivalent to: } A_s/A_i = [(m * (C_s/Amt_i)) + b]$$

where,

$C_s$	=	Concentration/amount of target analyte
$A_s$	=	Area for target analyte [e.g., PCB8]
$A_i$	=	Area for internal standard [e.g., PCB39]
$Amt_i$	=	Amount internal standard [e.g., PCB39 added to sample]
$W$	=	Sample size (g, dry wt)
$b$	=	y-intercept of linear regression equation.
$m$	=	slope of linear regression equation.

However, the ECD does not respond linearly and we typically calibrate with a quadratic equation for PCB target compounds. A quadratic equation was consistently used in this project (e.g. see curve type in method description on page 000073 of the bird and fish egg data package). The page references listed below for the example calculations are for the bird and fish egg GC/ECD data package.

The quadratic equation is considerably more complicated than the above listed linear regression equation, and takes a full page of calculation steps to perform. I do not think you want to subject yourself to that. We have carried out that exercise a few times to validate the data system. The method calibrates correctly as long as the correct standard amounts are put into the method (pages 000079 to 000081). The samples are correctly quantified as long as the correct recovery internal standard amounts are entered for the sample [e.g., see page 000282 where the appropriate recovery internal standards are listed with the ng amounts spiked for each sample as designated in the method]. The amount of RIS spiked into each sample can be traced to the sample preparation records [e.g. page 000013]. The two recovery internal standards are PCB39, which is used for congeners PCB1 through PCB119, and PCB166 which is used for congeners PCB112 through PCB209 (based on GC retention order and as listed on quantitation printouts).

The PCB amounts can be found on the *quantitation reports*. Quantitation reports are the data system generated reports which represent the analytes quantified with a given method, and report the result in as ng.

### Attachment 8 (cont.)

The PCB concentrations is calculated as follows:

$$[\text{PCB}] = \text{PCB Amount} * \text{Lipid}_{\text{SF}} * \text{Coplanar}_{\text{SF}} * \text{PIV}_{\text{SF}} * \text{Sample}_{\text{DF}} \times 1 / \text{Sample}_{\text{WT}}$$

[PCB] =	PCB concentration (ng/g, wet weight)
PCB Amount =	Amount of PCB in the sample analyzed on the GC instrument, as listed on the quantitation report (ng)
Lipid <sub>SF</sub> =	Lipid analysis subsampling factor. Factor that corrects for the amount removed in the lipid analysis. (total extract volume before lipid analysis) × (1 / (total extract volume before lipid analysis – extract volume removed for lipid analysis))
Coplanar <sub>SF</sub> =	Coplanar analysis split factor. Factor that corrects for any splitting of the extract for coplanar PCB analysis. (total extract volume before split / extract volume removed for the subject analysis)
PIV <sub>SF</sub> =	PIV subsampling factor. Factor that corrects for subsampling of the extract prior to the addition of RIS and submission for initial instrumental. This factor only applies to liver samples which had a volume removed from the concentrated extract, and the subsample was spiked with RIS prior to analysis. (total extract volume before subsampling / extract volume removed for the subject analysis)
Sample <sub>DF</sub> =	Sample dilution factor. Factor that corrects for any subsampling of the extract for dilution purposes (i.e., when samples were diluted and re-analyzed). This is only a factor when a portion of the extract is removed, and subsequently spiked with additional RIS. The amount of solvent added to perform the dilution is not a factor in the calculation. Additionally, this is <i>not</i> a factor if only the PIV is increased to bring the analyte response within the calibration range in a re-analysis. (total extract volume before subsampling / extract volume removed for the subject dilution and re-analysis)
Sample <sub>WT</sub> =	Sample weight (g, wet weight). Weight of the sample amount that was extracted.

**Example:**

Sample ID:	Z5799, page 000305 through 000307 - quantitation report (from batch 97-126), page 000439 and 000440 (97-126 Table)
Data File Number:	pesticides_chan1_01.sa06,21,1, page 000282 (# = 21)
PCB Amount, Target Analyte:	423.7752 ng, PCB28, page 000305
Lipid <sub>SF</sub> :	1.056, page 000010
Coplanar <sub>SF</sub> :	2, page 000012 (10000uL was initial volume, which was then split, 5000uL to each analysis)
PIV <sub>SF</sub> :	1, page 000013 (the RIS was added to the entire 5000uL)
Sample <sub>DF</sub> :	1, page 000013 (no dilution was performed on this sample)
Sample <sub>WT</sub> :	5.33g wet wt., page 000006

$$\text{PCB28 (ng/g)} = [(423.7752 * 1.056 * 2 * 1 * 1) / 5.33]$$

$$\text{PCB28 (ng/g)} = 167.92 \text{ ng/g (page 000439)}$$

Attachment 8 (cont.)

CALCULATION OF SURROGATE RECOVERY -- PCB by GC/ECD

Surrogate recoveries are also simply calculated using surrogate internal standard quantitation data obtained directly from the sample quantitation reports (the applicable peak areas and recovery internal standard amounts have already been incorporated into the method/equation for the quantitation report generation, as presented in the "Quantification of Samples" text). The surrogate internal standard determined amount listed on the quantitation report is a direct measure of the amount of surrogate internal standard recovered, using the quantitation method of our data system. The surrogate recoveries are calculated as detailed below:

$$SR = [(RS/Amt) \cdot Lipid_{SF} \cdot Coplanar_{SF} \cdot PIV_{SF} \cdot Sample_{DF} \cdot 100\%]$$

where,

Amt = Amount surrogate internal standard [e.g., PCB36] added to sample  
Lipid<sub>SF</sub> = As explained in the "Quantification of Samples" text  
Coplanar<sub>SF</sub> = As explained in the "Quantification of Samples" text  
PIV<sub>SF</sub> = As explained in the "Quantification of Samples" text  
Sample<sub>DF</sub> = As explained in the "Quantification of Samples" text  
RS = Amount surrogate internal standard [e.g., PCB36] determined in sample

The two surrogate internal standards used for surrogate recoveries are PCB36 and PCB112.

There are two recovery internal standards, PCB39 and PCB166. The recovery internal standard PCB39 is used to determine the recovery of the surrogate internal standard PCB36 and the recovery internal standard PCB166 is used to determine the recovery of the surrogate internal standard PCB112.

**Example:**

Sample ID: Z5799, pages 000305 through 000307 - quantitation report (from batch 97-126), page 000439 and 440 (97-126 Table)  
Data File Number: pesticides,chan1\_01.sa06,21,1, page 000282 (# = 21)  
Amt, Surrogate Internal Standard Spiked (ng): 803.2, page 000008 (400 uL EI17 \* 2.008 ng/uL)  
Lipid<sub>SF</sub>: 1.056, page 000010  
Coplanar<sub>SF</sub>: 2, page 000012 (10000uL was initial volume, which was then split, 5000uL to each analysis)  
PIV<sub>SF</sub>: 1, page 000013 (the RIS was added to the entire 5000uL)  
Sample<sub>DF</sub>: 1, page 000013 (no dilution was performed on this sample)  
RS, Surrogate Internal Standard Amount Determined (ng): 349.2781, page 000305

$$SR, PCB36 Recovery (\%) = [(349.2781/803.2) \cdot 1.056 \cdot 2 \cdot 1 \cdot 1 \cdot 100\%]$$

$$SR, PCB36 Recovery (\%) = 92 \text{ (page 000440)}$$



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August 26, 1997

Mr. Douglas Beltman  
Hagler Bailly Consulting, Inc  
1881 Ninth Street, #201  
Boulder, CO 80302

Subject: Reporting of PCB Data for the Lower Fox River/Green Bay NRDA Project — GC/MS Data

Dear Doug:

Enclosed please find Battelle data packages for tissue sample analyses performed in support of the *Lower Fox River/Green Bay NRDA Project*. These data are for the analyses of 26 tissue samples by GC/MS and the GC/ECD MDL samples, as described in the project-specific Quality Assurance Project Plan, dated May 5, 1997 and your supplemental memorandum dated July 22, 1997 which describes the selection of samples for GC/MS analysis. The samples were analyzed for the determination of polychlorinated biphenyl (PCB) concentrations.

The GC/MS data are reported in two large 3-ring binders. One binder contains the tern egg and fish egg PCB congener data, and the other binder contains the PCB congener data for the whole body walleye, brown trout, and lake trout samples selected for GC/MS analysis. A smaller 3-ring binder is also included in this deliverable. This binder contains the data associated with the GC/ECD MDL determination for the 106 base PCB congeners.

The final data are printed out as summary spreadsheet tables in the "Tables" section of the data packages. Enclosed you will also find (1) one diskette with the Excel spreadsheet files that contain the summary data tables, (2) a table summarizing the calculated GC/ECD MDLs (Attachment 1), (3) a table with representative GC/MS reporting limits (Attachment 2), and (4) example calculations to aid the validator when reviewing the GC/MS data packages (Attachment 3).

A separate Excel spreadsheet has been prepared with transposed GC/MS field sample data (file named "Field Sum ExtendedMS.xls"), per your request and discussions with Tom Gulbransen. All 26 field samples have been pulled together into one table in this file. These data have also been compiled into a single Access data base file that is provided on a separate diskette. There are no hard copies of the transposed Excel table or the Access file because of their large size. Additionally, it should be noted that, per our discussions, the transposed data and the Access file have only received a cursory review, and I strongly recommend that your staff carefully check them against the standard deliverable tables before they are used. The standard summary spreadsheets tables, which are those that are included in the Tables section of the data packages, are the primary deliverable; these tables have all been thoroughly reviewed and validated by our independent QA Unit, as well as by staff of the chemistry department.

### *Analytical Information*

The 26 samples for which GC/MS data are reported are a sub-set of the tissue samples that were processed and analyzed by GC/ECD. The GC/ECD data were reported on August 13, 1997 and that deliverable included the technical procedural information, and other general supporting information that are associated with all of these analyses.

### *General Quality Control and Other Information*

- MDL Data — GC/ECD. The MDL sample analyses are compiled in one data package (the smaller of the three 3-ring binders). The MDL study was performed in accordance with the EPA protocol set forth in 40 CFR 136 Appendix B *Method Detection Limit (MDL) Determination*, with seven replicate analyses being used. Summary MDL data tables have been prepared for MDLs calculated the following four different ways: (1) concentrations calculated on a wet weight basis and quantification versus the recovery internal standard (i.e., no surrogate compound correction), (2) concentrations calculated on a wet weight basis and with surrogate compound correction, (3) concentrations calculated on a dry weight basis and quantification versus the recovery internal standard, (4) concentrations calculated on a dry weight basis and with surrogate compound correction. The MDL data based on sample wet weight and without surrogate correction (which is the method used for all samples in this project) are also presented in Attachment 1.

The hatchery trout fillet that was used for the MDL study had measurable levels of PCB, as did all the hatchery samples analyzed in this project. Unfortunately, this had a significant negative impact on the results of the MDL study because there were higher levels of many PCB congeners in the sample to begin with than was added for the MDL determination. The sample used for an MDL study should ideally not contain any of the target compounds prior to fortification. Although the non-spiked sample matrix was also analyzed non-fortified (and in duplicate), it was not possible to background correct the data because the native concentrations were so high relative to the spiking levels.

The MDLs were generally in the 0.10 to 0.15 ng/g range for the PCB congeners that were not present in the tissue material to begin with, or present at very low concentrations (Attachment 1) — these PCB congeners best represent the "true" MDLs for the method. These MDLs are consistent with our past experience, which have typically generated wet weight MDLs in the 0.02-0.05 ng/g range when there has been no sample splitting (the MDL samples in this study had a split factor of 2) and when using a sample size of about 25 g (the average sample weight was about 11 g in this study).

The surrogate recoveries for the MDL samples ranged from 67 to 103%. There were no notable levels of PCB detected in the PB sample, and the PCB congener recoveries were near 100% in the BS sample for almost all target compounds; the apparent over-recovery of PCB41 in the BS (which is qualified with an "X") is due to coelution with coplanar congener #37 which was added to the sample at a significant level. These results indicate that the quality of the sample analyses were in control.

- RL Data — GC/MS. Examples of GC/MS reporting limits are tabulated in Attachment 2. Sample-specific RLs were used for qualifying the analytical data, and the RLs that were used for data reporting differ from those presented in Attachment 2 depending on sample-specific PIVs, dilution factors etc. The reporting limits are based on the PCB congener concentration in the low calibrations standard and are calculated as described in detail in the August 13, 1997 deliverable. PCB congeners could typically be confidently determined at concentrations well below the RLs, and uncensored data were reported for this work and qualified with a "J", as appropriate.
- Quantification and Reporting of Congeners #153 and 132 — GC/MS. Congeners #153 and #132 could not be separated in the GC/MS analysis of the whole body fish samples (analytical batches 97-190, 97-191, and 97-192), and are therefore reported as PCB153/132, indicating that the value represents the sum of these two congeners. In Hagler Bailly's original scope of work for this project separate data for these congeners was not expected, although Battelle was able to provide discrete data from the GC/ECD analysis. These congeners could be separated during the GC/MS analysis of the egg samples, and separate data are reported for those samples.
- X Qualifier for Congener #153 in CRM Samples — GC/MS. The CRM results for congener #153 have been qualified for the whole body fish samples (analytical batches 97-190, 97-191, and 97-192), because of the previously mentioned coelution of congeners #153 and #132. The CRM results for congener #153 are clearly elevated in these three samples, as compared to the CRM data in the two egg batches, which can be attributed to contributions from congener #132.
- Comparison of GC/MS and GC/ECD Total PCB Data. The data for the 26 project field samples for which both GC/ECD and GC/MS analysis was performed have been given a cursory review to assess the comparability. The GC/MS data suggest that there was interference with certain congeners in the GC/ECD analysis, although generally the comparability is quite good. The significant interference observed with congener #85 in the GC/ECD analysis (likely p,p'-DDE) was transparent to the GC/MS analysis, and the GC/MS data can be used to obtain more reliable values for congener #85. A comparison of the sum of the PCB congener values from the GC/ECD and GC/MS analyses provide good general comparability information. The average RPD in the sum of the PCB congeners determined by GC/MS and GC/ECD was just under 8% (using the sum of the congeners without congener #85 to represent the GC/ECD analysis). As could be expected, the greatest comparability was observed for the analytical batches where the GC/ECD analyst reported the "cleanest" baselines and minimal matrix contributions (the fish egg and last whole body fish batch — batches 97-129 and 97-192). The sum of the PCB congener concentrations were, on average, only 4 and 3% different, respectively, between the two analytical methods for these batches. Analytical batches with more complex GC/ECD matrix signals had somewhat greater differences in the data; the RPD in the sum of the PCB congeners averaged 11% for the bird egg batch (batch 97-126).
- Error in Calibration Standard Table in GC/ECD Congener Package. The spreadsheet table listing the calibration standard concentrations that was included in the GC/ECD data package for the egg samples had a few minor errors. The errors had been detected, the original spreadsheet updated in Battelle's standards records, but had not been updated in this data package. The correct standard concentrations were used in all sample quantification, so no data were affected. I am enclosing these updated pages and an additional copy that highlights where corrections were made. Please replace pages 48 and 49 in the Standard Preparation section of the GC/ECD data package that contains the bird and fish egg data with these two new pages.

**Specific Quality Control Information — GC/MS Analysis**

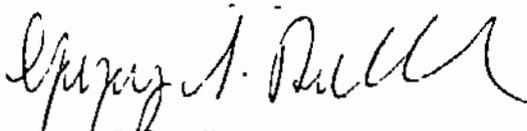
Analysis of procedural blanks was the only QC sample analysis that was required for the GC/MS work, as described in the project QAPP. Other QC samples data, and surrogate recovery information, were generated in the GC/ECD analyses. However, Battelle reduced the GC/MS data for the blank spike and certified reference material samples, and those data are included in the enclosed data packages.

- **Procedural (Method) Blanks.** A procedural blank (PB) was processed and analyzed with each of the five sample batches. There was no PCB detected in any of the PBs.
- **Blank Spike Recovery.** A blank spike (BS) sample was processed with each of the five analytical batches. Each of the 527 individual PCB congener recovery data points met the data quality objective, with the majority of the recoveries being in the 80 to 95% range.
- **CRM Recovery.** A certified reference material (CRM) was analyzed with each of the five analytical batches. This material is certified for selected PCB congeners. The CRM data are presented both non-corrected and surrogate corrected. The surrogate correction uses surrogate recoveries that were generated in the GC/ECD analysis because no surrogate recoveries were determined in the GC/MS analysis. It may not be appropriate to apply these GC/ECD surrogate recoveries to the GC/MS data since the target analytes (GC/MS data) and internal standards (GC/ECD data) may be impacted by different levels and types of analyte and matrix effects. The CRM data using non surrogate corrected GC/MS data probably provide the best data assessment, since surrogate recoveries were not determined in this analysis.

The average PD in the CRM results consistently met the DQO. The individual congener PD values also met the DQOs, even though the measured PCB170/190 concentration was below the primary target DQO ( $\pm 35$  PD for analytes with concentrations  $> 5$  times the RL) by 0.2% in one analysis (analytical batch 97-192); one analyte in each sample could be up to 50 PD from the certified value. The measured concentrations were typically 5 to 20 below the certified value, which is consistent with target analyte recoveries in the 80 to 95% range (as was observed for the BS samples).

Please do not hesitate to give me a call at 617-934-0571 if you have any questions at all.

Sincerely,



Gregory S. Durell  
Senior Research Scientist

**Attachments:**

- Attachment 1: MDLs for PCB Congeners by GC/ECD
- Attachment 2: PCB Analysis Reporting Limits — GC/MS
- Attachment 3: Example Data Calculations — GC/MS

## Attachment 1

## MDLs for PCB Congeners by GC/ECD

PCB Congener	MDL (ng/g, wet weight) *
PCB1	0.85
PCB3	2.42
PCB4/10	0.37
PCB7/9	0.15
PCB6	0.17
PCB8/5	0.23
PCB19	0.13
PCB12/13	0.93
PCB18	0.19
PCB17/15	0.15
PCB24/27	0.17
PCB16/32	0.24
PCB29	0.08
PCB26	0.14
PCB25	0.13
PCB31	0.30
PCB28	0.23
PCB21	0.10
PCB33/20	0.16
PCB53	0.20
PCB51	0.09
PCB22	0.09
PCB45	0.15
PCB46	0.16
PCB52	0.64
PCB43	0.11
PCB49	0.36
PCB47/75	0.27
PCB48	0.09
PCB44	0.19
PCB59	0.06
PCB42/37	0.35
PCB41/64/71	0.64
PCB40	0.18
PCB100	0.14
PCB63	0.21
PCB74	0.32
PCB70/76	0.34
PCB66	0.25
PCB95	0.53
PCB91	0.18
PCB56/60	0.17
PCB92	0.19
PCB84	0.42
PCB89	0.25
PCB101/90	0.95
PCB99	0.67
PCB119	0.15
PCB83	0.20
PCB97	0.42
PCB87/115/81	0.56
PCB85	1.49
PCB136	0.14

PCB Congener	MDL (ng/g, wet weight) <sup>a</sup>
PCB110/77	0.92
PCB82	0.22
PCB151	0.42
PCB135/144	0.29
PCB124	0.12
PCB107/147	0.21
PCB149/123	0.87
PCB118	0.99
PCB134	0.10
PCB114	1.62
PCB131	0.21
PCB146	0.43
PCB153	1.70
PCB132	0.70
PCB105	0.40
PCB141/179	0.22
PCB137	0.12
PCB176	0.14
PCB130	0.18
PCB138/160/163	1.33
PCB158	0.16
PCB129/126	0.14
PCB178	1.77
PCB175	0.12
PCB187/182	0.71
PCB183	0.26
PCB128	0.28
PCB167	0.15
PCB185	0.14
PCB174	0.21
PCB177	0.27
PCB171/202	0.28
PCB156	0.12
PCB173	0.13
PCB201/157	0.18
PCB172	0.12
PCB197	0.11
PCB180	1.49
PCB193	0.13
PCB191	0.13
PCB200	0.11
PCB169	0.80
PCB170/190	0.32
PCB198	0.09
PCB199	0.31
PCB203/196	0.26
PCB189	0.16
PCB195/208	0.28
PCB207	0.12
PCB194	0.28
PCB205	0.12
PCB206	0.42
PCB209	0.25

<sup>a</sup> Wet weight MDL values without surrogate compound correction. Average sample weight was 11.08 g, and the split factor was 2 (i.e., only half the sample was sent to analysis).

## Attachment 2

## REPORTING LIMITS - Extended PCB Congener Set by GC/MS

<b>Batch ID</b>	97-126	97-129	97-190, 97-191, 97-192	97-191, 97-192	97-192
<b>Matrix</b>	<i>Tern eggs</i>	<i>L. Trout Eggs</i>	<i>Walleye Whale</i>	<i>B. Trout Whale</i>	<i>L. Trout Whale</i>
Pre-Injection Volume (uL)	5000	2000	1000	2000	1000
Lipid Analysis Split Factor	1.00	1.00	1.00	1.00	1.00
Coplanar Analysis Split Factor	2.00	2.00	1.00	1.00	1.00
Sample Dilution Factor	1.00	1.00	1.00	1.00	1.00
Sample Wet Weight (g)	5.00	5.00	5.00	5.00	10.00
Reporting Unit	ng/g, wet weight	ng/g, wet weight	ng/g, wet weight	ng/g, wet weight	ng/g, wet weight
<b>Analyte</b>					
PCB1	102.6	41.0	10.3	20.5	5.1
PCB3	201.0	80.4	20.1	40.2	10.1
PCB4	102.8	41.1	10.3	20.6	5.1
PCB7	64.2	25.7	6.4	12.8	3.2
PCB6	64.0	25.6	6.4	12.8	3.2
PCB8	66.8	26.7	6.7	13.4	3.3
PCB19	36.0	14.4	3.6	7.2	1.8
PCB12	64.0	25.6	6.4	12.8	3.2
PCB18	33.4	13.4	3.3	6.7	1.7
PCB17	28.4	11.4	2.8	5.7	1.4
PCB24	36.0	14.4	3.6	7.2	1.8
PCB16	36.0	14.4	3.6	7.2	1.8
PCB29	36.0	14.4	3.6	7.2	1.8
PCB26	38.0	14.4	3.6	7.2	1.8
PCB25	35.8	14.3	3.6	7.2	1.8
PCB31	38.0	14.4	3.6	7.2	1.8
PCB28	33.2	13.3	3.3	6.6	1.7
PCB21	36.0	14.4	3.6	7.2	1.8
PCB33	36.0	14.4	3.6	7.2	1.8
PCB53	38.6	15.4	3.9	7.7	1.9
PCB51	24.4	9.8	2.4	4.9	1.2
PCB22	36.0	14.4	3.6	7.2	1.8
PCB45	30.8	12.3	3.1	6.2	1.5
PCB48	38.6	15.4	3.9	7.7	1.9
PCB52	33.4	13.4	3.3	6.7	1.7
PCB43	36.0	14.4	3.6	7.2	1.8
PCB49	38.4	15.4	3.8	7.7	1.9
PCB47	38.6	15.4	3.9	7.7	1.9
PCB48	38.6	15.4	3.9	7.7	1.9
PCB44	33.4	13.4	3.3	6.7	1.7
PCB59	38.4	15.4	3.8	7.7	1.9
PCB42	36.0	14.4	3.6	7.2	1.8
PCB41	29.6	11.8	3.0	5.9	1.5
PCB40	36.0	14.4	3.6	7.2	1.8
PCB100	38.6	15.4	3.9	7.7	1.9
PCB63	38.4	15.4	3.8	7.7	1.9
PCB74	38.6	15.4	3.9	7.7	1.9
PCB70	38.6	15.4	3.9	7.7	1.9
PCB66	33.4	13.4	3.3	6.7	1.7
PCB95	38.6	15.4	3.9	7.7	1.9
PCB91	29.6	11.8	3.0	5.9	1.5
PCB56	30.8	12.3	3.1	6.2	1.5
PCB92	27.0	10.8	2.7	5.4	1.4
PCB84	30.8	12.3	3.1	6.2	1.5
PCB89	38.4	15.4	3.8	7.7	1.9
PCB101	33.4	13.4	3.3	6.7	1.7
PCB99	38.4	15.4	3.8	7.7	1.9
PCB119	38.6	15.4	3.9	7.7	1.9
PCB83	38.4	15.4	3.8	7.7	1.9

REPORTING LIMITS - Extended PCB Congener Set by GC/MS

Batch ID Matrix	97-126 Tern eggs	97-129 L Trout Eggs	97-190, 97-191, 97-192 Walleye Whole	97-191, 97-192 B Trout Whole	97-192 L Trout Whole
PCB97	38.6	15.4	3.9	7.7	1.9
PCB87	38.6	15.4	3.9	7.7	1.9
PCB85	29.4	11.8	2.9	5.9	1.5
PCB136	38.6	15.4	3.9	7.7	1.9
PCB110	38.6	15.4	3.9	7.7	1.9
PCB82	38.6	15.4	3.9	7.7	1.9
PCB151	38.6	15.4	3.9	7.7	1.9
PCB135	28.2	11.3	2.8	5.6	1.4
PCB124	38.6	15.4	3.9	7.7	1.9
PCB107	38.6	15.4	3.9	7.7	1.9
PCB149	38.4	15.4	3.8	7.7	1.9
PCB118	33.4	13.4	3.3	6.7	1.7
PCB134	38.4	15.4	3.8	7.7	1.9
PCB114	38.6	15.4	3.9	7.7	1.9
PCB131	34.6	13.8	3.5	6.9	1.7
PCB146	28.4	11.4	2.8	5.7	1.4
PCB153	33.2	13.3	3.3	6.6	1.7
PCB132	38.6	15.4	3.9	7.7	1.9
PCB105	33.4	13.4	3.3	6.7	1.7
PCB141	38.6	15.4	3.9	7.7	1.9
PCB137	38.4	15.4	3.8	7.7	1.9
PCB178	30.8	12.3	3.1	6.2	1.5
PCB130	30.8	12.3	3.1	6.2	1.5
PCB138	33.2	13.3	3.3	6.6	1.7
PCB158	38.6	15.4	3.9	7.7	1.9
PCB129	38.6	15.4	3.9	7.7	1.9
PCB178	27.0	10.8	2.7	5.4	1.4
PCB175	38.6	15.4	3.9	7.7	1.9
PCB187	33.4	13.4	3.3	6.7	1.7
PCB183	33.4	13.4	3.3	6.7	1.7
PCB128	33.4	13.4	3.3	6.7	1.7
PCB167	38.6	15.4	3.9	7.7	1.9
PCB185	38.6	15.4	3.9	7.7	1.9
PCB174	24.4	9.8	2.4	4.9	1.2
PCB177	23.0	9.2	2.3	4.6	1.2
PCB171	38.5	15.4	3.9	7.7	1.9
PCB156	38.6	15.4	3.9	7.7	1.9
PCB173	38.4	15.4	3.8	7.7	1.9
PCB201	38.4	15.4	3.8	7.7	1.9
PCB172	38.4	15.4	3.8	7.7	1.9
PCB187	38.4	15.4	3.8	7.7	1.9
PCB180	33.4	13.4	3.3	6.7	1.7
PCB193	38.4	15.4	3.8	7.7	1.9
PCB191	38.6	15.4	3.9	7.7	1.9
PCB200	38.6	15.4	3.9	7.7	1.9
PCB169	38.6	15.4	3.9	7.7	1.9
PCB170	33.4	13.4	3.3	6.7	1.7
PCB199	38.4	15.4	3.8	7.7	1.9
PCB199	32.0	12.8	3.2	6.4	1.6
PCB203	30.8	12.3	3.1	6.2	1.5
PCB189	38.4	15.4	3.8	7.7	1.9
PCB195	33.4	13.4	3.3	6.7	1.7
PCB207	30.8	12.3	3.1	6.2	1.5
PCB194	38.4	15.4	3.8	7.7	1.9
PCB205	38.6	15.4	3.9	7.7	1.9
PCB206	25.6	10.2	2.6	5.1	1.3
PCB209	25.6	10.2	2.6	5.1	1.3

### Attachment 3

#### QUANTIFICATION OF SAMPLES – PCB by GC/MS

Samples are quantified using the method of internal standards. The quantification internal standard is the recovery internal standards (added to the sample immediately prior to instrumental analysis). The concentration of target analytes is determined using the following equation:

$$[\text{PCB}] = [(A/A_s) \times (\text{Amt}/\text{RF}_s) \times \text{Lipid}_{\text{SF}} \times \text{Coplanar}_{\text{SF}} \times \text{Sample}_{\text{DF}} \times (1/\text{Sample}_{\text{WT}})]$$

where,

[PCB]	=	Concentration target PCB analyte
A <sub>s</sub>	=	Area quantification ion for target analyte (e.g., PCB18)
A <sub>i</sub>	=	Area quantification ion for internal standard (PCB34)
Amt <sub>i</sub>	=	Amount internal standard (PCB34) added to sample
RF <sub>s</sub>	=	Average RF for analyte (e.g., PCB18) determined from initial calibration
Lipid <sub>SF</sub>	=	Lipid analysis subsampling factor. Factor that corrects for the amount removed in the lipid analysis. (total extract volume before lipid analysis) × (1/(total extract volume before lipid analysis – extract volume removed for lipid analysis))
Coplanar <sub>SF</sub>	=	Coplanar analysis split factor. Factor that corrects for any splitting of the extract for coplanar PCB analysis. (total extract volume before split / extract volume removed for the subject analysis)
Sample <sub>DF</sub>	=	Sample dilution factor. Factor that corrects for any subsampling of the extract for dilution purposes (i.e., when samples were diluted and re-analyzed). This is only a factor when a portion of the extract is removed, and subsequently spiked with additional RIS. The amount of solvent added to perform the dilution is not a factor in the calculation. Additionally, this is <i>not</i> a factor if only the PIV is increased to bring the analyte response within the calibration range in a re-analysis. (total extract volume before subsampling / extract volume removed for the subject dilution and re-analysis)
Sample <sub>WT</sub>	=	Sample weight (g, wet weight). Weight of the sample amount that was extracted.

One internal standard was used for quantification (PCB34).

Attachment 3 (cont.)

QUANTIFICATION OF SAMPLES – PCB by GC/MS (Continued)

The page references listed below for the example calculations are for the bird and fish egg GC/MS data package (analytical batches 97-126 and 97-129).

Example Calculation:

Sample ID: VA89CRM, page 000208 - 210, SQB347 pg 000107  
Data File Number: B0708.d, page 000208 - 210  
A, Target Analyte: PCB128, page 000210  
I, Internal Standard: PCB34, page 000208  
A<sub>t</sub>, Target Analyte Area: 1178, page 000210  
A<sub>i</sub>, Internal Standard Area: 11427, page 000208  
Amt<sub>i</sub>, Internal Standard Spike Amt (ng): 235, page 000208 and page 000076  
(100 μL of standard E119 × 2.35 ng/μL)  
Rf<sub>i</sub>, Average RF of PCB128: 0.56684, page 000113  
Lipids<sub>sf</sub>: 1.058, page 000073  
Coplanar<sub>sf</sub>: 2.0, page 000076  
Sample<sub>pf</sub>: 1.0, page 000076  
Sample<sub>wr</sub>: 5.03, page 000069

$$\text{PCB128 Conc. (ng/g)} = [(1178/11427) \times (235/0.56684) \times (1.058) \times (2) \times (1) \times (1/5.03)]$$

$$\text{PCB128 Conc. (ng/g)} = 14.24 \text{ ng/g (page 189)}$$

**Note:** Please review the information in the Miscellaneous Documentation section of the data package before beginning to audit and review data; this section may contain additional information that are important to the calculation of sample analyte concentrations.



December 5, 1997

Mr. Douglas Beltman  
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Subject: Reporting of PCB Data for the Lower Fox River/Green Bay NRDA Project —  
GC/ECD Data from Re-Quantified and Re-Analyzed Samples

Dear Doug:

Enclosed please find Battelle's data package for the *Lower Fox River/Green Bay NRDA Project* tissue sample analyses recently performed at Battelle Duxbury Operations. The samples were analyzed for the determination of polychlorinated biphenyl (PCB) concentrations. These data are from (1) the re-quantification of a set of samples that were originally quantified with a different calibration type, and (2) the re-extraction and re-analysis of a set of samples that had low surrogate recoveries in the original analysis. The original data for these samples, the data which you may wish to replace with these new data, were submitted as part of the large data delivery on August 13, 1997.

The GC/ECD data are reported in two large 3-ring binders, with the appropriate section dividers and tabs indicating the location of different data and information. The final data have been printed out as summary spreadsheet tables in the "Tables" section of the data package. Enclosed you will also find (1) one diskette with the Excel spreadsheet files that contain the summary data tables, (2) a table listing the samples that were re-quantified with the edited calibration method (Attachment 1), (3) a table listing the samples that were re-extracted and re-analyzed, and the types of analyses that was performed on them (Attachment 2), and (4) results of the lipid determination method comparison (Attachment 3). Enclosed is also a replacement page for one of the sample homogenization forms that were submitted earlier — the Battelle sample ID has been corrected for two samples (the correct ID was used in all sample preparation documentation and data deliveries).

Other relevant information such as the technical procedures, listing of target PCB analytes, data quality objectives, data qualifiers, reporting limits, method detection limits, example calculations, chain-of-custody documentation, etc. have been provided with previous data deliverables.

A sheet with transposed *field* sample data has been added to the two Excel spreadsheet files that contain field sample data (97-191 Requant\_a.xls and Re-extracts\_a.xls). The transposed data are in a format that can easily be accepted by data bases, and they have been compiled into an Access file (with three data tables). There are no hard copies of the transposed Excel tables or the Access file. Additionally, it should be noted that, per our discussions, the transposed data and the Access file have only received a cursory review, and I strongly recommend that your staff carefully check them against the standard deliverable tables before they are used. The standard summary spreadsheet tables, which are those included in the Tables section of the data package, are the primary deliverable; these tables have been thoroughly reviewed and validated by our QA Unit, as well as by staff of the chemistry department.

### *Analytical Information*

**Re-Quantified Samples.** Re-quantified extended PCB congener data are submitted for 21 samples (Attachment 1) for which data were already submitted back on August 13, 1997. The original data for these samples were inadvertently generated using a 1/X weighted quadratic equation, and the calibrations were therefore re-generated using a non-weighted method to be consistent with all the other data; non-weighted calibrations are also more standard. Most of the affected samples were QC samples because a large number of the field samples in those analytical batches were diluted and re-analyzed, and the re-analyses were quantified with a non-weighted calibration.

**Re-Analyzed Samples.** Three batches of re-extractions and re-analyses are reported in this data delivery (Attachment 2). These samples were re-analyzed in the laboratory because the recoveries were lower than desirable in the original analyses. Several samples from the first re-extraction batch (97-274) were actually re-analyzed a second time in one of the other two batches because the recoveries were still low. Additionally, the field samples in the second and third re-analysis batches were processed in duplicate to obtain the best data. Both replicates were reported if the surrogate recoveries were good for both analyses, and the better of the two was reported if one or both of the replicates yielded surrogate recoveries outside the data quality objective range.

High quality data were generated for most samples, but a few surrogate recoveries remained below the data quality objective, even though they were separately analyzed up to four (4) times, indicating unique sample matrix characteristics. However, considering the large numbers of samples analyzed and reported the overall quality of the project data set is very high and only three analyses (out of 175 separate PCB sample analyses) remain with recovery results below the data quality objectives.

### *General Quality Control and Other Information*

Several of the general reporting items listed in this section have already been communicated to Hagler Bailly, and are included here for completeness.

- **Re-Quantification.** The re-quantification of the samples listed in Attachment 1 yielded only slightly different data than what was submitted on August 13, 1997. For instance, samples VD38 (BTEG01CP) and VD40 (BTEG04CP) are now reported to have a "Sum of PCB w/o PCB85" concentration of 1,900 and 1,707 ng/g, wet weight, versus the original results of 1,955 and 1,752 ng/g, respectively. The new data for these samples are approximately 2-3% lower than the original results.
- **JX Qualifier for Congener #85.** There was significant coelution/interference with congener #85 in the field samples that appears to be caused by the presence of p,p'-DDE. Therefore, the congener #85 data have been qualified with the qualifier "JX" when this peak is clearly significantly higher than could reasonably be expected. The size of this peak was not considered when deciding on dilutions and re-analysis of samples (i.e., this peak was frequently above the high calibration standard and was often above the range of the detector, and ignored for dilution purposes). The data reported for congener #85 are not accurate, and, therefore, in addition to providing a sum of all PCB concentrations in the Excel summary tables, we are also providing a sum of all congeners with congener #85 excluded. The congener summation without congener #85 is likely a more accurate measure of the total PCB than the sum that includes congener #85.

For your information, congener #85 constitutes approximately 1% of the total PCB in mid-molecular weight Aroclor formulations, such as Aroclors 1248 and 1254 (Schultz *et al.*, 1989, *Environ. Sci. Technol.* 23, 852-859; and Battelle internal determinations). It is only reasonable to expect a similar contribution in environmental samples for this particular congener. The GC/MS data will provide more accurate congener #85 concentration data. The GC/MS data will also provide information on the relative ratio of congener #85 to other congeners that are not interfered with in the GC/ECD analysis, thus providing data that can be used to obtain a good estimate of the congener #85 concentrations in all samples.

- X Qualifier for Congener #169. There appeared to be a procedural contaminant in the coplanar PCB congener method that resulted in a doublet peak that interfered with congener #169; the two interfering peaks elute on either side of the congener #169 peak. If a peak was clearly present in the valley between the two contaminant it was picked as congener #169, but the contaminant most likely masked the presence of this congener under most circumstances or reduced the accuracy of any quantification of this congener when detected. The qualifier "X" was added to the congener #169 data in the coplanar analysis (whether it was detected or not) to indicate this issue.

Battelle followed EPA Method 1668 for the coplanar PCB congener separation, and it is unclear what reagent or other component of the method contributed this interference. Congener #169 was also determined in the standard PCB congener analysis (it is the only coplanar congener that can be well resolve in that analysis), and there was no evidence of procedural interference with congener #169 in those analyses.

- CRM Quantification. The quality control results for the Certified Reference Material (CARP-1 CRM) were calculated and reported *both* surrogate corrected and not surrogate corrected; separate spreadsheet tables have been prepared. The reason is that the certified values for this CRM are based on surrogate corrected quantification (per information from the National Research Council (NRC) Canada scientists who prepared and certified this material), and surrogate correction may therefore be the most valid approach for performing data comparisons.
- Quantification of Congener #63. There was an error in the concentration used for congener #63 in the calibration method for the second level of the multilevel calibration, and this was discovered after the samples had been quantified (0.0196 ng/ $\mu$ L was entered/used rather than the correct value of 0.0192). This minor error was for one analyte in one calibration level. A field sample was requantified with the correct concentration in the calibration method to assess the impact of this error. The two methods yielded result of 23.1886 and 23.1072 ng (<0.4% difference for congener #63, with the reported value being the higher of the two) and this relatively minor discrepancy for one congener was considered so small that it did not warrant re-quantification of the data set.
- Lipid Content Method Comparison. Lipid content determination was performed with two different extraction solvents (hexane and dichloromethane) on seven brown trout whole body samples and seven walleye whole body samples, to assess differences caused by the two solvents. Additionally, triplicate analysis was performed on one sample of each fish type. The results from this determination are summarized in Attachment 3.

As expected, the dichloromethane extraction method yielded higher lipid content values than the hexane extraction. The lipid content was, on average, about 43% higher with the dichloromethane method for brown trout and about 25% higher for walleye. However, these data need to be considered carefully

before they are used to generate some generic method-to-method lipid content correction factor because there is clearly significant fish-to-fish variability. The difference (percent difference) between the two methods was as low as 17% and as high as 72% for different brown trout samples, with the rest ranging from 39% to 50%. This notable fish-to-fish variability could be the result of slightly different lipid composition of different fish (i.e., the fish sample matrix). Additionally, and possibly even more importantly, variability in the *moisture* content of the fish impacts the variability in the lipid data when calculated on a wet weight basis; the lipid are primarily associated with the "dry" matrix, not the water. After normalizing the data for moisture content (i.e., calculating lipid content on a *dry*, not wet, weight basis) it is likely that the PD values will decrease. The precision in the triplicate analyses of the same sample is quite good, indicating that the observed variability is not due to the method.

#### *Specific Quality Control Information — Re-Analyzed Samples*

- **Procedural (Method) Blanks.** A procedural blank (PB) was processed and analyzed with each of the sample batches. Few congeners were detected in the PB samples in the extended PCB congener analysis, and those were consistently at very low levels — well below the reporting limits. In the coplanar PCB congener analyses there was interference with congener #169 (as discussed earlier) that contributed to a low-level signal in the PB, but none that suggested the presence of this congener. There was also a low-level signal corresponding to congener #81 in one of the three coplanar PCB congener PBs, but it too represented a concentration much below the reporting limits.
- **Blank Spike Recovery.** A blank spike (BS) sample was processed with each of the analytical batches. All extended congener target compound recoveries were acceptable for the BS sample. The BS recoveries were acceptable for the three coplanar PCB congener batches, with the exception of a slightly elevated recovery for congener #126 (135% recovery) in the BS processed with batch 97-306 and a slightly low recovery for congener #37 (48% recovery) in the BS with batch 97-312.
- **CRM Recovery.** A certified reference material (CRM) was analyzed with each of the analytical batches. This material is certified for selected "standard" PCB congeners (i.e., not for any coplanar congeners). For the coplanar PCB congener analyses the CRM was used only to track precision over several batches. CRM results are reported both non-corrected and surrogate corrected. The surrogate corrected results best represents the true native sample concentration and should be used for comparing with CRM certification values; surrogate corrected data were used by National Research Council (NRC) Canada when establishing the reference values.

The average PD in the CRM results met the DQO, and there was only one individual congener exceedance; 43 %PD for PCB66/95, versus a DQO of  $\pm 35\%$ . However, the CRM is not certified for PCB66/95 and a less rigorous "consensus" value is used to evaluate this parameter. The precision in the coplanar PCB analysis of the CRM was relatively good for congeners #77 and #126 (e.g., 32% RSD and 25% RSD, respectively, for the non-corrected data), considering the low concentrations of these congeners in the CRM (near or below the RL).

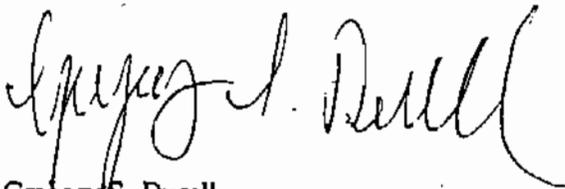
- **Duplicate (DUP) Precision.** Specific duplicate precision tables were not generated for all of the re-analyses because so many sets of replicate analyses were performed that no sample needed to be specifically designated as the DUP sample of the batch. A large number of replicate field samples are available to calculate analytical precision, and the template to "drop" such data in to are available as a separate sheet in the provided Excel files.

- Surrogate Recovery. Surrogate internal standard (SIS) compounds were added to every field and QC sample to monitor sample processing efficiency, and the recoveries of two SISs (congeners PCB36 and PCB112) were determined for each sample. Additionally, the coplanar PCB congener column separation efficiency was monitored using deuterated congener #77 for all samples that were subjected to coplanar PCB congener analysis.

The surrogate recoveries were generally very good. The surrogate recovery DQOs (50 to 125% recovery) were met for all QC samples and almost all field samples. As discussed earlier, a few field samples had surrogate recoveries that did not meet the DQOs even though they were separately analyzed up to four (4) times. This clearly indicates a unique sample matrix for these samples — a matrix that cannot be effectively extracted using standard laboratory procedures. However, considering the large numbers of samples that were analyzed in this project it is clear that the overall quality of the data set is very high and only three analyses remain with recovery results outside the DQO range. These three samples are all lake trout whole body samples: (1) the coplanar PCB congener data for sample Z6899 (EGLMF06WC-1) is based on a sample with low standard congener surrogate recoveries, (2) the coplanar PCB congener data for sample Z6833 (EGLMF10WC-1) is based on a sample with a low coplanar congener surrogate recovery, and (3) the extended PCB congener data for sample Z6834 (EGLMF12WC-1) is based on a sample with low standard congener surrogate recoveries. The low recoveries for these three ranged from 19% to 28%.

Please do not hesitate to give me a call at 781-934-0571 if you have any questions at all.

Sincerely,



Gregory S. Durell  
Senior Research Scientist

**Attachments:**

- Attachment 1: Re-Quantified Samples
- Attachment 2: Re-Analyzed Samples
- Attachment 3: Lipid Method Comparison

Attachment 1

Re-Quantified Samples<sup>a</sup>

Client ID	Battelle ID	Batch ID
NA	VD26PB	97-190
NA	VD27BS	97-190
NA	VD28CC	97-190
NA	VD29IB	97-190
NA	VD30EB	97-190
NA	VD32PB	97-191
NA	VD33BS	97-191
NA	VD34CC	97-191
NA	VD35IB	97-191
NA	VD36EB	97-191
BTEG01CP	VD38	97-191
BTEG04CP	VD40	97-191
BTUG01CP	VD42	97-191
BTUG02CP	VD43	97-191
BTUG04CP	VD44	97-191
BTUG05CP	VD45	97-191
NA	VD48PB	97-192
NA	VD49BS	97-192
NA	VD51CC	97-192
NA	VD52EB	97-192
NA	VD53IB	97-192

<sup>a</sup> Re-quantified by non-weighted quadratic calibration type because they were initially quantified with a 1/x weighted quadratic calibration. Target extended PCB congener data only (surrogate recoveries were not affected because they were not calibrated by the weighted method in the original analysis).

## Attachment 2

Re-Analyzed Samples <sup>a</sup>

Client Reporting ID	Battelle Sample ID <sup>b</sup>	Re-Analysis Batch ID	Original Batch ID	Congener Analysis Type <sup>c</sup>	Sample Type/Matrix
TEKIB18	Z5799	97-274	97-126	CP	Tern egg
96KICT05	Z5807	97-274	97-126	CP	Tern egg
96KICT07	Z5813	97-274	97-126	CP	Tern egg
96KICT09	Z5815	97-274	97-126	CP	Tern egg
WEFR07CP	VC59	97-274	97-192	CP	Walleye whole
WELG06CP	VC65	97-274	97-192	CP	Walleye whole
BTEG02CP	VD47	97-274	97-192	CP	B. trout whole body
EGLMF06WC-1	Z6899	97-274	97-192	CP	L. trout whole body
TEKIB48	Z5804	97-306	97-126	CP	Tern egg
96KICT03	Z5811	97-306	97-126	CP	Tern egg
96KICT10	Z5816	97-306	97-126	CP	Tern egg
WEWG04CP	VC69	97-306	97-192	CP	Walleye whole
EGLMF11WC-1	Z6897	97-306	97-192	CP	L. trout whole body
EGLMF07WC-1	Z6898	97-306	97-192	CP	L. trout whole body
EGLMF01FC-1	Z5958	97-306	97-129	STD	L. trout eggs
EGLMF08FC-1	Z5965	97-306	97-129	STD	L. trout eggs
EGLMF10WC-1	Z6833	97-306	97-192	CP + STD	L. trout whole body
EGLMF09WC-1	Z6901	97-306	97-192	CP + STD	L. trout whole body
EGLMF01WC-1	Z6902	97-312	97-192	CP	L. trout whole body
EGLMF02WC-1	Z6900	97-312	97-192	CP	L. trout whole body
EGLMF03WC-1	Z6881	97-312	97-192	CP	L. trout whole body
EGLMF04WC-1	Z6880	97-312	97-192	CP	L. trout whole body
EGLMF05WC-1	Z6879	97-312	97-192	CP	L. trout whole body
EGLMF08WC-1	Z6835	97-312	97-192	CP	L. trout whole body
EGLMF12WC-1	Z6834	97-312	97-192	CP	L. trout whole body
BTUG03CP	VD46	97-312	97-192	CP	B. trout whole body

<sup>a</sup> The listed field samples were re-extracted/re-analyzed with new QC samples (PB, BS, CRM, and DUP).

<sup>b</sup> The Battelle sample ID for the re-extracted and re-reported analyses has a -1 or -2 suffix as part of the ID to indicate if it is the first or second re-extraction/re-analysis of the sample. Additionally, all samples in batches 97-306 and 97-312 were re-analyzed in duplicate and the DUP designation has then also been added to the base Battelle ID for the sample tracking and data reporting (e.g., Z6901-2DUP). Both replicates were reported if both had surrogate recoveries that were well within the data quality objectives (DQOs). The sample with the better surrogate recoveries was reported if the recoveries were outside the DQOs for one or both replicates. Data for both replicates have been reported for Z5811, Z5816, VC69, Z6898, Z5958, Z5965, Z6901, Z6881, and VD46.

<sup>c</sup> Congener analysis type: CP; coplanar congeners. STD: standard extended list congeners.

LIPID METHOD COMPARISON - Hexane vs Dichloromethane

Client Reporting ID	Matrix	Battelle ID	Lipid Content (% wet weight)		PD
			Hexane as Solvent	DCM as Solvent	
BTEG01CP	Brown trout whole body	VD38	7.87	11.27	43.2
BTEG03CP	Brown trout whole body	VD39	8.12	12.16	49.8
BTEG04CP	Brown trout whole body	VD40	9.82	13.81	40.6
BTEG05CP	Brown trout whole body	VD41	11.74	13.75	17.1
BTUG01CP	Brown trout whole body	VD42	8.28	14.25	72.1
BTUG03CP	Brown trout whole body	VD46	10.75	15.38	43.1
BTEG02CP	Brown trout whole body	VD47	10.33	14.33	38.7
				Average:	43.5
				%RSD:	37.3
BTEG04CP	Brown trout whole body	VD47	10.33	14.33	
BTEG04CP	Brown trout whole body	VD47-DUP	9.46	13.27	
BTEG04CP	Brown trout whole body	VD47-TRIP	11.44	13.95	
		Average:	10.4	13.9	
		%RSD:	9.5	3.9	
WEFR04CP	Walleye whole body	VA57	8.94	11.42	27.7
WEFR01CP	Walleye whole body	VC53	8.59	9.58	11.5
WEFR02CP	Walleye whole body	VC54	14.56	17.19	18.1
WEFR03CP	Walleye whole body	VC55	9.63	10.79	12.0
WEFR05CP	Walleye whole body	VC57	13.52	16.72	23.7
WEFR06CP	Walleye whole body	VC58	11.81	16.90	43.1
WEFR07CP	Walleye whole body	VC59	12.59	17.84	41.7
				Average:	25.4
				%RSD:	51.1
WEFR07CP	Walleye whole body	VC59	12.59	17.84	
WEFR07CP	Walleye whole body	VC59-DUP	14.38	15.66	
WEFR07CP	Walleye whole body	VC59-TRIP	15.97	12.39	
		Average:	14.3	15.3	
		%RSD:	11.8	17.9	

Sample Homogenization

IR  
7/28/97 RLM

Project Name: Lower Fox River/Green Bay NRDA  
 Project Number: G003264  
 Homogenization Completed by: RLM  
 Homogenization method/equipment: Hobart Grinder  
 Storage Location Removed from: F1218  
 Storage Location until homogenization/compositing: CH-440  
 Storage Location Returned to: F1220

Date: 7/7/97  
 Date/Time: 7/7/97 8:00 AM  
 Date/Time: 7/7/97 5:00 PM

Sample Matrix	Sample #	Field Sample ID *	Battelle ID (log-in) *
Lake trout whole body	1	EGLMF01WC-1	Z6902
Lake trout whole body	2	EGLMF02WC-1	Z6900
Lake trout whole body	3	EGLMF03WC-1	Z6881
Lake trout whole body	4	EGLMF04WC-1	Z688280
Lake trout whole body	5	EGLMF05WC-1	Z688379
Lake trout whole body	6	EGLMF06WC-1	Z6899
Lake trout whole body	7	EGLMF07WC-1	Z6898
Lake trout whole body	8	EGLMF08WC-1	Z6835
Lake trout whole body	9	EGLMF09WC-1	Z6901
Lake trout whole body	10	EGLMF10WC-1	Z6833
Lake trout whole body	11	EGLMF11WC-1	Z6897
Lake trout whole body	12	EGLMF12WC-1	Z6834

XIO  
11/24/97  
RLM

\* The client Field Sample ID is the same as the Client Reporting ID for samples that are not composited, as outlined in Attachment 2 of the Project Laboratory QAPP. Similarly, the Battelle ID given at log-in is the same as the Battelle Reporting ID for samples that are not composited, such as those listed above.

# DATA VALIDATION REPORT

## PCB Analysis of Biota Tissues Green Bay Natural Resource Damage Assessment

### Prepared for:

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### Prepared by:

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EcoChem Project No.: C9309-3

April 10, 1998

### Approved for Release:

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Senior Chemist  
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## Introduction

This report summarizes the quality assurance evaluations performed and data qualifications recommended for 123 tissue samples analyzed for the Green Bay Natural Resource Damage Assessment project. Refer to the Sample Index (TABLE 1) for sample identifications and analyses.

The tissue samples were analyzed for 106 PCB congeners or seven Aroclor formations using the Battelle laboratory standard operating procedure, *Identification and Quantitation of Polychlorinated Biphenyls (by Congener and Aroclor) and Chlorinated Pesticides by Gas Chromatography/Electron Capture Detection*. Several samples that were analyzed for the standard congener list were also analyzed for five coplanar PCB congeners. A subset of 26 samples that were analyzed for the standard congener list were also analyzed by GC/MS. The analyses were performed by Battelle Ocean Sciences, 397 Washington Street, Duxbury, Massachusetts.

The surrogate percent recoveries for many of the samples were not within the acceptance limits in the initial analysis. Additionally, two sample extracts were spilled during the extraction process. For these two reasons, four samples for the standard congener analyses and 24 samples for the coplanar congener analyses were re-extracted and reanalyzed. The original results were qualified as do-not-report (DNR); the results from the re-extracted analyses should be used.

The primary data validation review was performed by Sherri Wunderlich and secondary technical review was performed by Alison Bodkin. The data validation review was based on the quality control criteria specified in the analytical methods and the data quality objectives listed in the QAPP.

Data validation and reasons for qualification are summarized in each section of the following report. Validation qualifier definitions and reason codes are listed in TABLE 2 AND TABLE 3, respectively. All data validation qualifiers appear in the database.

## FULL DATA VALIDATION REPORT

### PCB Analyses

Batches: 97-124, 97-126, 97-127, 97-128, 97-129, 97-181, 97-190, 97-191, 97-192, 97-274, 97-306, and 97-312

#### I. Data Package Completeness: ACCEPTABLE/With the following discussion.

All necessary documentation for the full validation were provided by the laboratory.

The chain-of-custody (COC) forms for six samples (96-KI-CT-01, 96-KI-CT-03, 96-KI-CT-05, 96-KI-CT-07, 96-KI-CT-09, and 96-KI-CT-10) did not list sampling dates.

For ten samples, EG-LM-F-01-FC-1 through EG-LM-F-10-FC-1, the collection date listed on the COC forms was 10/22/95. The laboratory indicated that the collection date listed on the sample bottles was 10/22/96. Since the COC forms were signed by the sampler on 10/22/96, and the COC collection date for the other two samples in the batch (Samples EG-LM-F-11-FC-1 and EG-LM-F-12-FC-1) was 10/22/96, the actual collection date was most likely 10/22/96.

Although, several internal sample custody seals were broken when received by the laboratory, all cooler seals were intact. This was probably caused by the pressure of the ice, as paper custody seals and tape can be weakened by the cold and moisture. No action was taken.

#### II. Sample Holding Times and Handling Conditions: ACCEPTABLE/With the following exceptions.

**Qualified Data:** See the Qualified Sample Results.

##### **Discussion:**

All tissue samples were stored frozen at -20 °C or below until the time of extraction. All samples were extracted within one year of the sampling date.

The analysis holding time criterion for PCBs is 40 days from extraction date to date of analysis. All samples were analyzed within the required holding time.

##### **Batch 97-192:**

**Sample BTUGO3CP:** During the extraction, approximately 30mL out of 200mL (or 15% of the sample extract volume) was spilled. As internal standardization was used to quantify the PCB congener concentrations, and as the standard extended list surrogate recoveries for this sample were acceptable, no qualification was performed based on the spillage.

**Sample EG-LM-F-09-WC-1:** During the preparation step, the vial containing the extract broke in the centrifuge. The sample was pipetted out of the rotor and put in a new vial. The laboratory re-

extracted and reanalyzed the sample by GC/ECD upon request. The reanalysis was performed with Batch 97-306. The original results were qualified as do-not-report (DNR-14); the results from the reanalysis should be used instead.

### III. Calibration: ACCEPTABLE/With the following exceptions.

**Qualified Data:** See the Qualified Sample Results.

#### **Discussion:**

##### **Initial Calibrations**

For the GC/ECD congener and Aroclor initial calibrations, all reported coefficients of determination for the initial calibrations were greater than or equal to 0.9900. (Therefore, correlation coefficients were greater than or equal to 0.9950.) The laboratory incorrectly calculated calibration curve coefficients using 1/X weighted values for three congener Aroclor initial calibrations (that were analyzed on 7/15/97, 7/22/97 and 8/8/97). The laboratory submitted corrections for the 7/15/97 and 7/22/97 initial calibrations and recalculated all associated sample results. For example, for Sample BTEG01CP the PCB sum (without PCB 85) was originally reported as 1955 ng/g versus a new total of 1900 ng/g; this represents a percent difference of less than 3%. The 8/8/97 initial calibration was only associated with the method detection limit study. As the weighted results were only slightly different than the non-weighted results, the method detection limit study was judged as not significantly affected.

For the GC/MS initial calibrations, all percent relative standard deviation (%RSD) values were less than the 35% upper control limit. All relative response factor values were greater than the 0.050 lower control limit.

##### **Continuing Calibrations (CCVs)**

Several percent difference (%D) results (from the true values) for target analytes were outside the individual compound control limit of  $\pm 25\%$ . Positive sample results that were associated with non-compliant %D values were qualified as estimated (J-5B). Non-detect results were judged to be not significantly affected. Qualified results are summarized in **TABLE 4**.

Several samples were not analyzed within 12 hours of the beginning CCV. Since all samples were bracketed by acceptable beginning and ending CCVs, no action was taken.

### IV. Blank Analyses: ACCEPTABLE/With the following exceptions.

**Qualified Data:** See the Qualified Sample Results.

#### **Discussion:**

Several PCB congeners were detected at low levels by the GC/ECD in some of the procedural, instrument, and equipment blanks. Action levels were established at five times the reported blank

concentrations. Associated positive sample results less than the action levels were qualified as not detected (U-7). Qualified results are summarized in **TABLE 5**.

**V. Surrogate Recovery:** ACCEPTABLE/With the following exceptions.

**Qualified Data:** See the Qualified Sample Results.

**Discussion:**

Several surrogate percent recovery (%R) values were outside the 50% to 125% control limits. The %R outliers are summarized in **TABLE 6**, sample results qualified as a result of surrogate outliers are summarized in **TABLE 7**, and specific details are provided in the following text.

**Standard Congener Analysis (GC/ECD)**

For Sample WEEG04CP (Batch 97-191), one surrogate %R value was outside of the control limits. A laboratory duplicate analysis of this sample was performed, with acceptable %R values. The result from the field sample was qualified as do not report (DNR-13); the results from the laboratory duplicate should be used.

Sample EG-LM-F-01-FC-1 in Batch 97-129, and Samples EG-LM-F-09-WC-1 and EG-LM-F-10-WC-1 in Batch 97-192 were re-extracted and reanalyzed due to unacceptable surrogate recoveries. Sample EG-LM-F-09-WC-1 was also reanalyzed due to the sample spilling in the extraction process; see **SECTION II**. Sample EG-LM-F-08-FC-1 (Batch 97-129) had surrogate recovery values that were slightly above the lower control limit of 50% at 53% for Surrogate PCB 63 and 51% for Surrogate PCB 112. Although these recoveries were technically within the control limits, the sample was re-extracted and reanalyzed to verify the recovery values. These re-extractions and reanalyses resulted in acceptable %R values. The results from the original analyses were qualified as do not report (DNR-13); the results from the reanalyses should be used.

**Coplanar Congener Analysis (GC/ECD)**

Seven samples in Batch 97-126 and 17 samples in Batch 97-192 (summarized in **TABLE 8**) were re-extracted and reanalyzed because of low surrogate percent recoveries. The results from the original analyses were qualified as do not report (DNR-13); the results from the reanalyses should be used.

For all other field samples summarized in **TABLE 7**, results were qualified as estimated (J-13/UJ-13) for %R values less 50% but greater than or equal to 10%. For %R values greater than the upper control limit, positive results were qualified as estimated (J-13); reporting limits were judged as not affected. Qualifiers were not assigned to QC samples.

Surrogate %R values less than the control limit may indicate that the sample results are biased low. The reported sample results are potentially underestimated. Surrogate %R values greater than the control limit indicate that the sample results are potentially biased high; however,

analytical interferences may be present that impact only the surrogate compounds, and these interferences may not impact the sample results.

#### ***GC/MS Analysis***

As indicated in the QAPP, surrogate %R values were not calculated for the GC/MS analyses. Surrogates were evaluated based on %R values obtained from the GC/ECD analyses. The GC/MS sample results were qualified as estimated (J-13) when the recovery values from the GC/ECD analyses were not within the control limits.

The surrogate %R value ranges for all batches are summarized in **TABLE 9**.

#### **VI. Blank Spike Sample Analysis: ACCEPTABLE/With the following exceptions.**

***Qualified Data:*** See the **Data Qualifier Summary Table**.

##### ***Discussion:***

A blank spike (BS) was extracted and analyzed at the frequency requirement of one per batch. All spiked analyte recovery values were within the control limits of 50% to 125% for tri- through deca-chlorobiphenyls and 30% to 125% for mono- and dichlorobiphenyls, with the exceptions listed in **TABLE 10**.

Results associated with BS recovery values that were less than control limits were qualified as estimated (J-10/UJ-10). Positive results associated with BS recovery values that were greater than control limits were qualified as estimated (J-10). See **TABLE 11** for a summary of results qualified because of blank spike and SRM outliers.

The blank spike %R value ranges for all analytes within a batch are listed in the **TABLE 12**.

#### **VII. Sample Duplicate Analysis: ACCEPTABLE/With the following exceptions.**

***Qualified Data:*** See the **Qualified Sample Results**.

##### ***Discussion:***

One or more duplicate samples were extracted with each batch. The duplicate sample was analyzed by GC/ECD, but not GC/MS (as specified in the work plan). Several relative percent difference (RPD) values were greater than the control limit of 50% as listed in **TABLE 13**.

All associated sample results were qualified as estimated (J-9), with the exception of the results associated with the GC/ECD Extended PCB Congener laboratory duplicate analysis performed on Sample EG-LM-F-01-FC-1 (Batch 97-129). As mentioned in **SECTION V**, the surrogate %R values were less than the lower control limit for this field sample, but acceptable in the laboratory duplicate. The target analyte concentrations for positive results were likewise much lower in the field sample; thus, the RPD values were greater than 50%. Since the field sample was already qualified for surrogate recoveries, and the low recoveries were attributed to an isolated incident

(not indicative of a systematic problem for the batch), no qualifiers were assigned due to laboratory duplicate results for Batch 97-129. Qualified results are summarized in **TABLE 14**.

**VIII. Standard Reference Material (SRM) Analysis:** ACCEPTABLE/With the following exceptions.

**Qualified Data:** See the Qualified Sample Results.

**Discussion:**

SRM Carp-1 samples (acquired from the National Research Council, Canada) were extracted and analyzed at the required frequency of one per each batch. The results for the SRM were calculated and reported both surrogate-corrected and not surrogate-corrected; separate spreadsheet tables were submitted. Since the certified values for this SRM are based on surrogate-corrected quantification, only the surrogate-corrected results were evaluated for the GC/ECD analyses. The GC/ECD SRM surrogate recovery value ranges were 82% to 115% (PCB 36) and 61% to 92% (PCB 112) for all sample batches. For the GC/MS analyses, only the uncorrected values were evaluated because the surrogate-corrected values were based on surrogate %R values from the GC/ECD analyses. All results were within the established acceptance criteria, with the exceptions listed in **TABLE 15**.

For reported values that were greater than the upper acceptance criterion, positive results in associated samples were qualified as estimated (J-10). For reported values that were less than the lower acceptance criterion, associated sample results were qualified as estimated (J-10/UJ-10). See **Table 11** for a summary of results qualified because of blank spike and SRM outliers.

**IX. Compound Identification and Quantitation:** ACCEPTABLE/With the following exceptions.

**Qualified Data:** See the Qualified Sample Results.

**Discussion:**

As discussed in the **Calibrations Section**, several standard congener sample results were originally calculated incorrectly (using incorrect initial calibration coefficients) for the GC/ECD analyses. The laboratory submitted corrected results for the following samples: four QC samples for Batch 97-190 (the procedural blank, instrument blank, equipment blank, and blank spike); four QC samples (the procedural blank, instrument blank, equipment blank, and blank spike) and six field samples for Batch 97-191 (Samples BTEG01CP, BTEG04CP, BTUG01CP, BTUG02CP, BTUG04CP, and BTUG05CP); and four QC samples for Batch 97-192 (the procedural blank, instrument blank, equipment blank, and blank spike).

**Standard Congener Analysis (GC/ECD)**

The laboratory stated that there was significant coelution/interference with PCB85 in the field samples, which appeared to be caused by the presence of p,p'-DDE. Positive results for PCB85 may be biased high. All positive results for PCB85 were qualified as estimated (J-14). Since the

results for PCB85 may not be accurate, the laboratory provided a sum of all congeners with PCB85 excluded (as well as a sum of all congeners with PCB85 included). The congener summation without PCB85 is likely to be the more accurate measure of the total PCBs.

#### *Coplanar Congener Analysis (GC/ECD)*

The laboratory stated that there was a contaminant interfering with PCB169. The interference was a doublet peak on each side of the PCB169 peak. If a peak was clearly present in the valley between the two contaminant peaks, it was identified as PCB169, but the contaminant most likely masked the presence of this congener or reduced the accuracy of any quantification of this congener when detected. PCB169 results from the co-planar analyses were qualified as estimated (J-14/UJ-14).

#### *Aroclor Analysis (GC/ECD)*

The chromatograms of the walleye liver samples closely resembled a mixture of Aroclors 1248 and 1254. The results were reported as "1248.1254." The PCB pattern in the trout fillets most closely resembled Aroclor 1254, and results were reported to reflect this identification.

#### *GC/MS Analysis*

The laboratory assigned ME and MI qualifiers to several PCB22 and PCB16 results to reflect estimated positive results and estimated reporting limits, respectively. The laboratory stated that a matrix interference was present. The ME lab qualifier was applied in situations where the primary ion profile displayed somewhat of a bell-shaped curve but contained obvious saturation, while the secondary ion profile was present and clearly displayed a bell-shaped profile. The MI lab qualifier was applied when both the primary and secondary ions did not show bell-shaped profiles, or when the primary and secondary ions did not show bell-shaped profiles at the same retention time. All sample results that were flagged ME or MI by the laboratory were qualified as estimated (J-14/UJ-14). Qualified results are summarized in **Table 16**.

#### **X. GC/ECD and GC/MS Results Comparison: ACCEPTABLE/With the following discussion.**

The results of the 26 samples that were analyzed by both GC/MS and GC/ECD for standard congeners are summarized in **Table 17**. As discussed in **SECTION IX**, there was significant coelution/interference with PCB85 in the field samples for the GC/ECD standard congener analyses, which appeared to be caused by the presence of p,p'-DDE. Since the results for PCB85 are biased high for the GC/ECD analyses, the sum of all congeners with PCB85 excluded were used to compare to the GC/MS results. (For the GC/ECD analyses, the congener summation without PCB85 is likely to be a more accurate measure of the total PCB than the sum that includes PCB85. The GC/MS data provides a more accurate quantitation of PCB85.)

The RPD values for results from the GC/MS and GC/ECD analyses were all less than 20% indicating acceptable precision between the methods.

**XI. Lipids Analysis:** ACCEPTABLE/With the following discussion.

For each batch (excluding the re-extracted batches), percent lipids were performed in duplicate for one sample. For Batch 97-129, the percent lipid RPD values between the original and duplicate results were greater than the control limit of 20% at 43.5%. No qualification of data was necessary as the two lipid values were relatively low (the difference was 1.19%).

For Batch 197-128, the percent lipid RPD values between the original and duplicate results were greater than the control limit of 20.0% at 20.5%. For Batch 97-181, the percent lipid RPD values between the original and duplicate results were greater than the control limit at 28.3%. For Batch 97-192, the percent lipid RPD values between the original and duplicate results were greater than the control limit at 38.6%. Although these percent lipid results for these three batches were not qualified the data user should be aware of potential bias as a result of a lack of homogeneity. All other sample/duplicate percent lipid RPD values were less than the upper control limit of 20.0%.

All RPD values for consecutive weighings were less than the upper control limit of 20.0%.

***Comparison of Solvents on % Lipid Values***

The laboratory originally selected three samples for a comparison of lipid content using different solvents (hexane and dichloromethane). For two of the sample sets, the dichloromethane extraction method yielded %D values (dichloromethane relative to hexane) of 38.4% to 40.7% higher lipid content values. For the third sample set, the lipid content was 6% higher with the dichloromethane solvent. The sample amounts used for the comparison test were relatively small (5.10 to 7.46 grams for the hexane solvent and 0.9987 to 1.0354 grams for the dichloromethane solvent). The laboratory performed the comparison study on more samples, in order to obtain more statistically-reliable results.

The laboratory selected seven brown trout whole body samples and seven walleye whole body samples for another comparison study. The dichloromethane extraction method yielded higher lipid content values than the hexane extraction. The average lipid content was 43.5% higher with the dichloromethane method than the hexane method for the brown trout samples and 25.4% higher for the walleye samples.

The laboratory stated that the data are to be considered carefully before they are used to generate a generic method-to-method lipid content correction factor because there is clearly significant fish-to-fish variability. The %D values between the two methods ranged from 17.1% to 72.1% for the trout and 11.5% to 43.1% for the walleye. This notable fish-to-fish variability could be the result of slightly different lipid composition of different fish. Additionally, variability in the moisture content of the fish impacts the variability in the lipid data when calculated on a wet weight basis; the lipid are primarily associated with the dry matrix, not the wet. If the data were normalized for moisture content (i.e., calculating lipid content on a dry, not wet, weight basis), it is likely that the %D values between the methods will decrease.

Triplicate analyses were performed on one brown trout and one walleye sample. The %RSD values ranged from 3.9% to 17.9%, and were judged as acceptable, indicating that the observed variability between the different solvents is not due to the method.

**XII Moisture Analysis:** ACCEPTABLE/With the following discussion.

For each batch (excluding the re-extracted batches), percent moisture content was performed in duplicate for one sample. For Batch 97-124, the percent lipid RPD values between the original and duplicate results were greater than the control limit of 20.0% at 38.8%. For Batch 97-126, the percent lipid RPD values between the original and duplicate results were greater than the control limit at 30.8%. The laboratory stated that the percent moisture for the duplicate sample was performed several weeks after the original percent moisture. No qualifiers were assigned on this basis. All other RPD values were less than the upper control limit of 20.0%.

All RPD values for consecutive weighings were less than the upper control limit of 20.0%.

**XIII. Overall Assessment of the Data**

Based on this evaluation, the laboratory followed the specified method.

Accuracy was generally acceptable, as demonstrated by the %R values of the surrogate, the blank spike, and the SRM analytes, except where previously noted. Precision was generally acceptable, as demonstrated by the RPD values of the sample and laboratory duplicates, except where previously noted.

Qualifiers were assigned due to blank contamination, CCV %D outliers, blank spike results, surrogate outliers, laboratory duplicate results, SRM Carp-I results, and chromatographic interferences.

Data that are qualified as DNR should not be used. All other data, as qualified, are acceptable for use.

**Table 1**  
**SAMPLE INDEX**  
**CLIENT: HAGLER BAILLY**  
**PROJECT NAME: GREEN BAY NRDA PROJECT**  
**EcoCHEM PROJECT No.: C9309-3**

Sample ID	Aroclors by GC/ECD	Standard Congeners by GC/ECD	Co-Planar Congeners by GC/ECD	Standard Congeners by GC/MS
WEFR01LV	✓			
WELG04LV	✓			
WELG03LV	✓			
WELG02LV	✓			
WEWG02LV	✓			
WEWG04LV	✓			
WEEG04LV	✓			
WEEG02LV	✓			
WEEG01LV	✓			
WEUG01LV	✓			
WEUG02LV	✓			
WEUG03LV	✓			
TE-K1-B-06		✓	✓	✓
TE-K1-B-18		✓	✓	
TE-K1-B-24		✓	✓	
TE-K1-B-30		✓	✓	
TE-K1-B-48		✓	✓	
TE-K1-B-60		✓	✓	✓
96-KI-CT-01		✓	✓	✓
96-KI-CT-03		✓	✓	
96-KI-CT-05		✓	✓	
96-KI-CT-07		✓	✓	
96-KI-CT-09		✓	✓	
96-KI-CT-10		✓	✓	✓
BT-EG-01-FC-1	✓			
BT-EG-03-FC-1	✓			
BT-EG-04-FC-1	✓			
BT-EG-05-FC-1	✓			
BT-EG-06-FC-1	✓			
BT-EG-07-FC-1	✓			
BT-EG-09-FC-1	✓			
BT-GA-01-FC-1	✓			
BT-GA-02-FC-1	✓			
BT-GA-03-FC-1	✓			

Table 1  
**SAMPLE INDEX**  
 CLIENT: HAGLER BAILLY  
 PROJECT NAME: GREEN BAY NRDA PROJECT  
 ECOCHEM PROJECT No.: C9309-3

Sample ID	Aroclors by GC/ECD	Standard Congeners by GC/ECD	Co-Planar Congeners by GC/ECD	Standard Congeners by GC/MS
BT-GA-04-FC-1	✓			
BT-GA-05-FC-1	✓			
LT-LM-01-FC-1	✓			
LT-LM-02-FC-1	✓			
LT-LM-03-FC-1	✓			
LT-LM-04-FC-1	✓			
LT-LM-05-FC-1	✓			
LT-LM-06-FC-1	✓			
LT-LM-07-FC-1	✓			
LT-LM-08-FC-1	✓			
LT-LM-09-FC-1	✓			
LT-LM-10-FC-1	✓			
LT-IR-02-FC-1	✓			
LT-IR-06-FC-1	✓			
LT-IR-07-FC-1	✓			
EG-LM-F-01-F-C-1		✓	✓	
EG-LM-F-02-F-C-1		✓	✓	✓
EG-LM-F-03-F-C-1		✓	✓	
EG-LM-F-04-F-C-1		✓	✓	
EG-LM-F-05-F-C-1		✓	✓	✓
EG-LM-F-06-F-C-1		✓	✓	
EG-LM-F-07-F-C-1		✓	✓	
EG-LM-F-08-F-C-1		✓	✓	
EG-LM-F-09-F-C-1		✓	✓	✓
EG-LM-F-10-F-C-1		✓	✓	✓
EG-LM-F-11-F-C-1		✓	✓	
EG-LM-F-12-F-C-1		✓	✓	
LT-IR-08-FC-1	✓			
BT-EG-02-FC-1	✓			
BT-EG-08-FC-1	✓			
WELG01LV	✓			
WEWG01LV	✓			
WEWG03LV	✓			
WEEG03LV	✓			

**Table 1**  
**SAMPLE INDEX**  
**CLIENT: HAGLER BAILLY**  
**PROJECT NAME: GREEN BAY NRDA PROJECT**  
**ECO-CHEM PROJECT NO.: C9309-3**

Sample ID	Aroclors by GC/ECD	Standard Congeners by GC/ECD	Co-Planar Congeners by GC/ECD	Standard Congeners by GC/MS
WEUG04LV	✓			
LT-IR-01-FC-1	✓			
WEFR01CP		✓		
WEFR02CP		✓		
WEFR03CP		✓		✓
WEFR04CP		✓		
WEFR05CP		✓		
WEFR06CP		✓		
WELG02CP		✓		
WELG03CP		✓		
WELG04CP		✓		
WELG05CP		✓		
WEWG01CP		✓		
WEWG02CP		✓		✓
WEWG03CP		✓		✓
WEEG01CP		✓		
WEEG03CP		✓		
WEEG04CP		✓		
WEEG05CP		✓		
WEEG06CP		✓		
WEEG07CP		✓		✓
WEEG08CP		✓		
WEEG10CP		✓		✓
WEEG11CP		✓		
WEUG01CP		✓		✓
WEUG03CP		✓		✓
BTEG01CP		✓		
BTEG03CP		✓		
BTEG04CP		✓		
BTEG05CP		✓		✓
BTUG01CP		✓		
BTUG02CP		✓		✓
BTUG04CP		✓		
BTUG05CP		✓		✓

**Table 1**  
**SAMPLE INDEX**  
**CLIENT: HAGLER BAILLY**  
**PROJECT NAME: GREEN BAY NRDA PROJECT**  
**ECO-CHEM PROJECT No.: C9309-3**

Sample ID	Aroclors by GC/ECD	Standard Congeners by GC/ECD	Co-Planar Congeners by GC/ECD	Standard Congeners by GC/MS
WEFR07CP		✓	✓	✓
WELG06CP		✓	✓	✓
WEEG04CP		✓	✓	
WEEG09CP		✓	✓	
WEUG02CP		✓	✓	
BTUG03CP		✓	✓	
BTEG02CP		✓	✓	✓
EG-LM-F-10-WC-1		✓	✓	✓
EG-LM-F-12-WC-1		✓	✓	
EG-LM-F-08-WC-1		✓	✓	
EG-LM-F-05-WC-1		✓	✓	✓
EG-LM-F-04-WC-1		✓	✓	
EG-LM-F-03-WC-1		✓	✓	✓
EG-LM-F-11-WC-1		✓	✓	
EG-LM-F-07-WC-1		✓	✓	
EG-LM-F-06-WC-1		✓	✓	
EG-LM-F-02-WC-1		✓	✓	
EG-LM-F-09-WC-1		✓	✓	✓
EG-LM-F-01-WC-1		✓	✓	
WELG01CP		✓		✓
WEEG02CP		✓		

**Table 2**  
**DATA VALIDATION QUALIFIER DEFINITIONS**

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
DNR	Do not report. A more usable set of data should be used instead.

**Table 3**  
**DATA VALIDATION QUALIFIER CODES**

1	Holding Times
2	Sample Preservation
3	Sample Custody
4	Missing Deliverables
5A	Calibration (initial)
5B	Calibration (continuing)
6	Field Blanks
7	Laboratory Blanks
8	Matrix Spike
9	Precision (Duplicate, or Matrix Spike Duplicate)
10	Laboratory Control Sample
11	Detection Limit
12	Standards
13	Surrogates
14	Other
15	Furnace QC
16	ICP Serial Dilution
17	Chemical Recoveries
18	Trip Blanks
19	Internal Standards
20	Linear Range Exceeded
21	Potential False Positives

**Table 4**  
**SAMPLE RESULTS QUALIFIED AS A RESULT OF CONTINUING CALIBRATION OUTLIERS**

**Table 5**  
**SAMPLE RESULTS QUALIFIED AS A RESULT OF BLANK OUTLIERS**

**Table 6**  
**SURROGATE PERCENT RECOVERY OUTLIERS**

Batch ID	Sample ID	Analysis	Congener	Percent Difference Value
97-124	97-124 CRM	GC/ECD Aroclor	PCB36	46%
	97-124 CRM	GC/ECD Aroclor	PCB112	35%
	WELG03LV	GC/ECD Aroclor	PCB112	127%
97-126	TE-K1-B-18	GC/ECD Congener Coplanar	PCB77-d	38%
	TE-K1-B-48	GC/ECD Congener Coplanar	PCB77-d	46%
	96-K1-CT-03	GC/ECD Congener Coplanar	PCB77-d	44%
	96-K1-CT-05	GC/ECD Congener Coplanar	PCB77-d	14%
	96-K1-CT-07	GC/ECD Congener Coplanar	PCB77-d	8%
	96-K1-CT-09	GC/ECD Congener Coplanar	PCB77-d	16%
	96-K1-CT-10	GC/ECD Congener Coplanar	PCB77-d	47%
97-128	97-128 CRM	GC/ECD Aroclor	PCB112	42%
97-129	EG-LM-F-01-FC-1	GC/ECD Congener (Standard)	PCB36	23%
	EG-LM-F-01-FC-1	GC/ECD Congener (Standard)	PCB112	17%
97-190	WEFR03CP	GC/ECD Congener (Standard)	PCB36	127%
	97-190 EB	GC/ECD Congener (Standard)	PCB112	126%
97-191	WEEG04CP	GC/ECD Congener (Standard)	PCB36	133%
	WEEG05CP	GC/ECD Congener (Standard)	PCB36	146%
	WEEG07CP	GC/ECD Congener (Standard)	PCB36	127%
97-192	WELG06CP	GC/ECD Congener (Standard)	PCB112	128%
	WEEG09CP	GC/ECD Congener (Standard)	PCB36	134%
	WEUG02CP	GC/ECD Congener (Standard)	PCB36	138%
	EG-LM-F-10-WC-1	GC/ECD Congener (Standard)	PCB36	178%
	EG-LM-F-10-WC-1	GC/ECD Congener (Standard)	PCB112	135%
	EG-LM-F-07-WC-1	GC/ECD Congener (Standard)	PCB112	48%
	EG-LM-F-09-WC-1	GC/ECD Congener (Standard)	PCB36	151%
	EG-LM-F-09-WC-1	GC/ECD Congener (Standard)	PCB112	137%
	WEFR07CP	GC/ECD Congener Coplanar	PCB77-d	17%
	WEFR06CP	GC/ECD Congener Coplanar	PCB77-d	33%
	WEFR04CP	GC/ECD Congener Coplanar	PCB77-d	48%
	BTU03CP	GC/ECD Congener Coplanar	PCB77-d	37%
	BTEG02CP	GC/ECD Congener Coplanar	PCB77-d	40%
	EG-LM-F-10-WC-1	GC/ECD Congener Coplanar	PCB77-d	13%
	EG-LM-F-12-WC-1	GC/ECD Congener Coplanar	PCB77-d	10%
	EG-LM-F-08-WC-1	GC/ECD Congener Coplanar	PCB77-d	29%
	EG-LM-F-05-WC-1	GC/ECD Congener Coplanar	PCB77-d	10%
	EG-LM-F-04-WC-1	GC/ECD Congener Coplanar	PCB77-d	10%
	EG-LM-F-03-WC-1	GC/ECD Congener Coplanar	PCB77-d	32%

**Table 6**  
**SURROGATE PERCENT RECOVERY OUTLIERS**

Batch ID	Sample ID	Analysis	Congener	Percent Difference Value
	EG-LM-F-11-WC-1	GC/ECD Congener Coplanar	PCB77-d	21%
	EG-LM-F-07-WC-1	GC/ECD Congener Coplanar	PCB77-d	17%
	EG-LM-F-06-WC-1	GC/ECD Congener Coplanar	PCB77-d	19%
	EG-LM-F-02-WC-1	GC/ECD Congener Coplanar	PCB77-d	16%
	EG-LM-F-09-WC-1	GC/ECD Congener Coplanar	PCB77-d	33%
	EG-LM-F-01-WC-1	GC/ECD Congener Coplanar	PCB77-d	44%
	WEFR07CP DUP	GC/ECD Congener Coplanar	PCB77-d	43%
	97-192 BS	GC/ECD Congener Coplanar	PCB77-d	136%
97-274	WEFR07CP	GC/ECD Congener Coplanar	PCB112	47%
	WEFR07CP DUP	GC/ECD Congener Coplanar	PCB112	43%
	WELG06CP	GC/ECD Congener Coplanar	PCB112	42%
	TEKIB18	GC/ECD Congener Coplanar	PCB112	42%
	96KICT05	GC/ECD Congener Coplanar	PCB112	39%
	96KICT07	GC/ECD Congener Coplanar	PCB112	32%
	96KICT09	GC/ECD Congener Coplanar	PCB112	40%
	EGLMF06WC-1	GC/ECD Congener Coplanar	PCB36	21%
	EGLMF06WC-1	GC/ECD Congener Coplanar	PCB112	19%
97-306	97-306 BS	GC/ECD Congener Coplanar	PCB77-d	134%
	EG-LM-F-10-WC-1	GC/ECD Congener Coplanar	PCB77-d	20%
97-312	EG-LM-F-12-WC-1	GC/ECD Congener Coplanar	PCB36	28%
	EG-LM-F-12-WC-1	GC/ECD Congener Coplanar	PCB112	24%

**Table 7**  
**SAMPLE RESULTS QUALIFIED AS A RESULT OF SURROGATE PERCENT RECOVERY**  
**OUTLIERS**

**Table 8**  
**SAMPLES RE-EXTRACTED AND REANALYZED FOR COPLANAR ANALYSIS**

<b>Batch 97-126:</b>	TE-KI-B-18 96-KI-CT-07	TE-KI-B-48 96-KI-CT-09	96-KI-CT-03 96-KI-CT-10	96-KI-CT-05
<b>Batch 97-192:</b>	WEFR07CP BTEG02CP EG-LM-F-04-WC-1 EG-LM-F-08-WC-1 EG-LM-F-12-WC-1	WELG06CP EG-LM-F-01-WC-1 EG-LM-F-05-WC-1 EG-LM-F-09-WC-1	WEWG04CP EG-LM-F-02-WC-1 EG-LM-F-06-WC-1 EG-LM-F-10-WC-1	BTUG03CP EG-LM-F-03-WC-1 EG-LM-F-07-WC-1 EG-LM-F-11-WC-1

**Table 9**  
**SURROGATE PERCENT RECOVERY RANGES**

<b>Batch ID</b>	<b>Analysis</b>	<b>Surrogate Range</b>
97-124	GC/ECD Aroclors	83% - 127%
97-126	GC/ECD Congener (Standard)	72% - 110%
	GC/ECD Congener (Coplanar)	8%* - 87%
97-127	GC/ECD Aroclors	73%-107%
97-128	GC/ECD Aroclors	61% - 121%
97-129	GC/ECD Congener (Standard)	17%* - 97%
	GC/ECD Congener (Coplanar)	84% - 120%
97-181	GC/ECD Aroclors	57% - 100%
97-190	GC/ECD Congener (Standard)	63% - 127%
97-191	GC/ECD Congener (Standard)	61% - 146%
97-192	GC/ECD Congener (Standard)	48%* - 178%
	GC/ECD Congener (Coplanar)	10%* - 78%
97-274	GC/ECD Congener (Coplanar)	19%* - 123%
97-306	GC/ECD Congener (Standard)	64% - 105%
	GC/ECD Congener (Coplanar)	20%* - 125%
97-312	GC/ECD Congener (Coplanar)	24%* - 108%

\*As a result of these low surrogate recoveries, the samples were re-extracted and reanalyzed (See SECTION V).

**Table 10  
BLANK SPIKE PERCENT DIFFERENCE OUTLIERS**

<b>Batch ID</b>	<b>Analysis</b>	<b>Analyte</b>	<b>Percent Difference Value</b>
97-126	GC/ECD Congener (Standard)	PCB175	44%
97-190	GC/ECD Congener (Standard)	PCB87	130%
	GC/ECD Congener (Standard)	PCB176	198%
	GC/ECD Congener (Standard)	PCB169	128%
97-191	GC/ECD Congener (Standard)	PCB176	139%
	GC/ECD Congener (Standard)	PCB169	151%
97-192	GC/ECD Congener (coplanar)	PCB169	142%
97-306	GC/ECD Congener (coplanar)	PCB126	135%
97-312	GC/ECD Congener (coplanar)	PCB37	48%

Table 11  
SAMPLE RESULTS QUALIFIED AS A RESULT OF BLANK SPIKE PERCENT RECOVERY  
OUTLIERS

**Table 12  
BLANK SPIKE PERCENT RECOVERY RANGES**

<b>Batch ID</b>	<b>Analysis</b>	<b>Blank Spike %R Range</b>
97-124	GC/ECD Aroclors	79% - 81%
97-126	GC/ECD Congener (Standard)	44% - 114%
	GC/ECD Congener (Coplanar)	63% - 84%
	GC/MS Congener (Standard)	79% - 98%
97-127	GC/ECD Aroclors	72% - 74%
97-128	GC/ECD Aroclors	75% - 75%
97-129	GC/ECD Congener (Standard)	51% - 109%
	GC/ECD Congener (Coplanar)	66% - 95%
	GC/MS Congener (Standard)	69% - 109%
97-181	GC/ECD Aroclors	75% - 76%
97-190	GC/ECD Congener (Standard)	81% - 196%
	GC/MS Congener (Standard)	77% - 90%
97-191	GC/ECD Congener (Standard)	75% - 151%
	GC/MS Congener (Standard)	77% - 100%
97-192	GC/ECD Congener (Standard)	68% - 91%
	GC/ECD Congener (Coplanar)	95% - 142%
	GC/MS Congener (Standard)	69% - 91%
97-274	GC/ECD Congener (Coplanar)	80% - 103%
97-306	GC/ECD Congener (Standard)	73% - 93%
	GC/ECD Congener (Coplanar)	86% - 135%
97-312	GC/MS Congener (Coplanar)	48% - 68%

**Table 13  
DUPLICATE RELATIVE PERCENT DIFFERENCE OUTLIERS**

Batch ID	Analysis	Sample	Analyte	RPD Value
97-126	GC/ECD Congener (Standard)	TE-KI-B-06	PCB63	74.6%
			PCB132	72.1%
	GC/ECD Congener (coplanar)		PCB37	87.6%
			PCB81	59.5%
97-129	GC/ECD Congener (Standard)	EG-LM-F-01-FC-1	PCB85	156.5%
			PCB110/77	144.3%
			PCB118	149.7%
			PCB153	170.5%
			PCB105	140.4%
			PCB138/160/163	157.4%
	GC/ECD Congener (coplanar)		PCB126	146.0%
			PCB77	169.2%
97-191	GC/ECD Congener (Standard)	WEEG04CP	PCB85	56.3%
97-192	GC/ECD Congener (Standard)	WEFR07CP	PCB85	51.2%
			PCB180	72.4%
97-274	GC/ECD Congener (coplanar)	WEFR07CP	PCB126	58.6%
97-306	GC/ECD Congener (Standard)	EG-LM-F-01-FC-1	All positive results >MDL except PCB114	> 50%
		EG-LM-F-08-FC-1	All positive results >MDL	> 50%

**Table 14**  
**SAMPLE RESULTS QUALIFIED AS A RESULT OF DUPLICATE RELATIVE PERCENT**  
**OUTLIERS**

**Table 15**  
**STANDARD REFERENCE MATERIAL OUTLIERS**

<b>Batch ID</b>	<b>Analysis</b>	<b>Analyte</b>	<b>Acceptance Criteria (ng/g)</b>	<b>Reported Value (ng/g)</b>
97-126	GC/ECD Congener (Standard)	PCB 18	13.8 - 28.8	12.56
	GC/ECD Congener (Standard)	PCB 170/190	14.3 - 29.7	10.67
97-129	GC/ECD Congener (Standard)	PCB 18	13.8 - 28.8	12.02
	GC/ECD Congener (Standard)	PCB 187/182	23.4 - 48.6	22.57
	GC/ECD Congener (Standard)	PCB 180	29.9 - 62.1	29.39
	GC/ECD Congener (Standard)	PCB 170/190	14.3 - 29.7	11.06
97-190	GC/ECD Congener (Standard)	PCB 66/95	87.1 - 180.9	191.06
	GC/ECD Congener (Standard)	PCB 118	85.8 - 178.2	269.25
	GC/ECD Congener (Standard)	PCB 153	54.0 - 112.1	138.86
	GC/MS Congener (GC/MS)	PCB 128	11.0 - 22.8	10.9
	GC/MS Congener (GC/MS)	PCB 170/190	14.3 - 29.7	13.7
97-191	GC/ECD Congener (Standard)	PCB 66/95	87.1 - 180.9	185.79
	GC/ECD Congener (Standard)	PCB 118	85.8 - 178.8	295.00
	GC/ECD Congener (Standard)	PCB 153	54.0 - 112.1	160.71
	GC/ECD Congener (Standard)	PCB 138/163/164	66.3 - 137.7	157.21
	GC/ECD Congener (Standard)	PCB 180	29.9 - 62.1	68.53
97-192	GC/ECD Congener (Standard)	PCB 118	85.8 - 178.2	246.30
97-306	GC/ECD Congener (Standard)	PCB 66/95	87.1 - 180.9	191.87

**Table 16**  
**GC/MS RESULTS QUALIFIED AS A RESULT OF POTENTIAL MATRIX INTERFERENCE**

Table 17  
GC/ECD - GC/MS SAMPLE RESULT RPD RANGES

## SAMPLE RESULTS QUALIFIED AS A RESULT OF BLANK SPIKE AND SRM OUTLIERS (J/UJ-10)

Client_ID	Batch_ID	Matrix	Parameter	Concentration (ng/g, wet)
96KICT01	97-126	Tern Eggs	PCB18	1.54676
96KICT01	97-126	Tern Eggs	PCB175	6.67678
96KICT01	97-126	Tern Eggs	PCB170/190	168.53827
96KICT03	97-126	Tern Eggs	PCB18	0.00000
96KICT03	97-126	Tern Eggs	PCB175	1.91263
96KICT03	97-126	Tern Eggs	PCB170/190	54.94693
96KICT05	97-126	Tern Eggs	PCB18	0.00000
96KICT05	97-126	Tern Eggs	PCB175	2.10017
96KICT05	97-126	Tern Eggs	PCB170/190	73.09801
96KICT07	97-126	Tern Eggs	PCB18	0.00000
96KICT07	97-126	Tern Eggs	PCB175	1.38117
96KICT07	97-126	Tern Eggs	PCB170/190	73.61305
96KICT09	97-126	Tern Eggs	PCB18	1.13160
96KICT09	97-126	Tern Eggs	PCB175	2.70641
96KICT09	97-126	Tern Eggs	PCB170/190	52.57390
96KICT10	97-126	Tern Eggs	PCB18	0.00000
96KICT10	97-126	Tern Eggs	PCB175	3.32402
96KICT10	97-126	Tern Eggs	PCB170/190	116.38861
TEKIB06	97-126	Tern Eggs	PCB175	4.43473
TEKIB06	97-126	Tern Eggs	PCB18	5.49921
TEKIB06	97-126	Tern Eggs	PCB170/190	104.89890
TEKIB18	97-126	Tern Eggs	PCB18	1.92204
TEKIB18	97-126	Tern Eggs	PCB175	3.34540
TEKIB18	97-126	Tern Eggs	PCB170/190	59.55321
TEKIB24	97-126	Tern Eggs	PCB175	3.51152
TEKIB24	97-126	Tern Eggs	PCB18	3.83493
TEKIB24	97-126	Tern Eggs	PCB170/190	60.34811
TEKIB30	97-126	Tern Eggs	PCB175	0.00000
TEKIB30	97-126	Tern Eggs	PCB18	2.97246
TEKIB30	97-126	Tern Eggs	PCB170/190	53.32203
TEKIB48	97-126	Tern Eggs	PCB18	2.09935
TEKIB48	97-126	Tern Eggs	PCB175	2.66088
TEKIB48	97-126	Tern Eggs	PCB170/190	45.96529
TEKIB60	97-126	Tern Eggs	PCB18	3.24269
TEKIB60	97-126	Tern Eggs	PCB175	7.42168
TEKIB60	97-126	Tern Eggs	PCB170/190	133.98202
EGLMF02FC-1	97-129	Lake Trout Eggs	PCB18	0.20411
EGLMF02FC-1	97-129	Lake Trout Eggs	PCB187/182	11.26807
EGLMF02FC-1	97-129	Lake Trout Eggs	PCB180	22.98183
EGLMF02FC-1	97-129	Lake Trout Eggs	PCB170/190	5.32721
EGLMF03FC-1	97-129	Lake Trout Eggs	PCB18	0.63694
EGLMF03FC-1	97-129	Lake Trout Eggs	PCB187/182	4.94289
EGLMF03FC-1	97-129	Lake Trout Eggs	PCB180	10.82764
EGLMF03FC-1	97-129	Lake Trout Eggs	PCB170/190	2.42365
EGLMF04FC-1	97-129	Lake Trout Eggs	PCB18	0.93536
EGLMF04FC-1	97-129	Lake Trout Eggs	PCB187/182	9.85506
EGLMF04FC-1	97-129	Lake Trout Eggs	PCB180	18.21514
EGLMF04FC-1	97-129	Lake Trout Eggs	PCB170/190	4.48064
EGLMF05FC-1	97-129	Lake Trout Eggs	PCB18	1.93279
EGLMF05FC-1	97-129	Lake Trout Eggs	PCB187/182	13.36881

## SAMPLE RESULTS QUALIFIED AS A RESULT OF BLANK SPIKE AND SRM OUTLIERS (J/UJ-10)

Client_ID	Batch_ID	Matrix	Parameter	Concentration (ng/g, wet)
EGLMF05FC-1	97-129	Lake Trout Eggs	PCB180	23.85250
EGLMF05FC-1	97-129	Lake Trout Eggs	PCB170/190	6.58034
EGLMF06FC-1	97-129	Lake Trout Eggs	PCB18	0.00000
EGLMF06FC-1	97-129	Lake Trout Eggs	PCB187/182	12.11489
EGLMF06FC-1	97-129	Lake Trout Eggs	PCB180	23.40895
EGLMF06FC-1	97-129	Lake Trout Eggs	PCB170/190	5.29166
EGLMF07FC-1	97-129	Lake Trout Eggs	PCB18	0.99927
EGLMF07FC-1	97-129	Lake Trout Eggs	PCB187/182	11.07883
EGLMF07FC-1	97-129	Lake Trout Eggs	PCB180	23.14357
EGLMF07FC-1	97-129	Lake Trout Eggs	PCB170/190	5.34569
EGLMF09FC-1	97-129	Lake Trout Eggs	PCB18	1.71783
EGLMF09FC-1	97-129	Lake Trout Eggs	PCB187/182	13.80636
EGLMF09FC-1	97-129	Lake Trout Eggs	PCB180	25.55064
EGLMF09FC-1	97-129	Lake Trout Eggs	PCB170/190	6.97092
EGLMF10FC-1	97-129	Lake Trout Eggs	PCB18	1.93920
EGLMF10FC-1	97-129	Lake Trout Eggs	PCB187/182	14.27640
EGLMF10FC-1	97-129	Lake Trout Eggs	PCB180	27.61000
EGLMF10FC-1	97-129	Lake Trout Eggs	PCB170/190	7.35160
EGLMF11FC-1	97-129	Lake Trout Eggs	PCB18	1.26226
EGLMF11FC-1	97-129	Lake Trout Eggs	PCB187/182	4.86918
EGLMF11FC-1	97-129	Lake Trout Eggs	PCB180	15.22107
EGLMF11FC-1	97-129	Lake Trout Eggs	PCB170/190	2.57759
EGLMF12FC-1	97-129	Lake Trout Eggs	PCB18	0.66631
EGLMF12FC-1	97-129	Lake Trout Eggs	PCB187/182	8.77747
EGLMF12FC-1	97-129	Lake Trout Eggs	PCB180	20.02061
EGLMF12FC-1	97-129	Lake Trout Eggs	PCB170/190	4.13528
WEEG01CP	97-190	Walleye Whole	PCB66	633.49489
WEEG01CP	97-190	Walleye Whole	PCB95	148.70538
WEEG01CP	97-190	Walleye Whole	PCB87/115/81	97.68867
WEEG01CP	97-190	Walleye Whole	PCB118	353.89500
WEEG01CP	97-190	Walleye Whole	PCB153	493.99963
WEEG01CP	97-190	Walleye Whole	PCB169	4.15763
WEEG03CP	97-190	Walleye Whole	PCB66	661.00181
WEEG03CP	97-190	Walleye Whole	PCB95	153.82531
WEEG03CP	97-190	Walleye Whole	PCB87/115/81	116.20330
WEEG03CP	97-190	Walleye Whole	PCB118	331.75705
WEEG03CP	97-190	Walleye Whole	PCB153	460.58435
WEEG03CP	97-190	Walleye Whole	PCB176	3.97052
WEEG03CP	97-190	Walleye Whole	PCB169	4.79962
WEFR01CP	97-190	Walleye Whole	PCB66	374.96763
WEFR01CP	97-190	Walleye Whole	PCB95	92.56827
WEFR01CP	97-190	Walleye Whole	PCB87/115/81	42.24894
WEFR01CP	97-190	Walleye Whole	PCB118	107.28683
WEFR01CP	97-190	Walleye Whole	PCB153	76.24384
WEFR01CP	97-190	Walleye Whole	PCB169	0.30085
WEFR02CP	97-190	Walleye Whole	PCB66	342.07992
WEFR02CP	97-190	Walleye Whole	PCB95	80.43008
WEFR02CP	97-190	Walleye Whole	PCB87/115/81	37.63168
WEFR02CP	97-190	Walleye Whole	PCB118	98.74544
WEFR02CP	97-190	Walleye Whole	PCB153	73.34976

## SAMPLE RESULTS QUALIFIED AS A RESULT OF BLANK SPIKE AND SRM OUTLIERS (J/UJ-10)

Client_ID	Batch_ID	Matrix	Parameter	Concentration (ng/g, wet)
WEFR02CP	97-190	Walleye Whole	PCB176	1.13528
WEFR02CP	97-190	Walleye Whole	PCB169	0.43976
WEFR03CP	97-190	Walleye Whole	PCB66	444.70152
WEFR03CP	97-190	Walleye Whole	PCB95	105.24487
WEFR03CP	97-190	Walleye Whole	PCB87/115/81	56.57259
WEFR03CP	97-190	Walleye Whole	PCB118	132.92940
WEFR03CP	97-190	Walleye Whole	PCB153	106.43431
WEFR03CP	97-190	Walleye Whole	PCB176	1.62581
WEFR03CP	97-190	Walleye Whole	PCB169	0.86217
WEFR04CP	97-190	Walleye Whole	PCB66	239.63492
WEFR04CP	97-190	Walleye Whole	PCB95	50.30005
WEFR04CP	97-190	Walleye Whole	PCB87/115/81	44.05128
WEFR04CP	97-190	Walleye Whole	PCB118	153.53534
WEFR04CP	97-190	Walleye Whole	PCB153	133.95748
WEFR04CP	97-190	Walleye Whole	PCB176	0.54151
WEFR04CP	97-190	Walleye Whole	PCB169	0.34923
WEFR05CP	97-190	Walleye Whole	PCB66	373.18192
WEFR05CP	97-190	Walleye Whole	PCB95	98.29816
WEFR05CP	97-190	Walleye Whole	PCB87/115/81	46.37570
WEFR05CP	97-190	Walleye Whole	PCB118	108.33469
WEFR05CP	97-190	Walleye Whole	PCB153	79.74663
WEFR05CP	97-190	Walleye Whole	PCB176	0.75765
WEFR06CP	97-190	Walleye Whole	PCB66	479.89904
WEFR06CP	97-190	Walleye Whole	PCB95	88.36056
WEFR06CP	97-190	Walleye Whole	PCB87/115/81	58.34962
WEFR06CP	97-190	Walleye Whole	PCB118	145.70553
WEFR06CP	97-190	Walleye Whole	PCB153	124.89046
WEFR06CP	97-190	Walleye Whole	PCB176	1.61128
WEFR06CP	97-190	Walleye Whole	PCB169	1.10415
WELG02CP	97-190	Walleye Whole	PCB66	216.00470
WELG02CP	97-190	Walleye Whole	PCB95	52.68318
WELG02CP	97-190	Walleye Whole	PCB87/115/81	27.77964
WELG02CP	97-190	Walleye Whole	PCB118	65.49555
WELG02CP	97-190	Walleye Whole	PCB153	48.94966
WELG02CP	97-190	Walleye Whole	PCB176	0.72470
WELG02CP	97-190	Walleye Whole	PCB169	0.30559
WELG03CP	97-190	Walleye Whole	PCB66	304.97027
WELG03CP	97-190	Walleye Whole	PCB95	55.66528
WELG03CP	97-190	Walleye Whole	PCB87/115/81	63.26913
WELG03CP	97-190	Walleye Whole	PCB118	215.47620
WELG03CP	97-190	Walleye Whole	PCB153	193.67681
WELG03CP	97-190	Walleye Whole	PCB176	0.89256
WELG03CP	97-190	Walleye Whole	PCB169	0.56876
WELG04CP	97-190	Walleye Whole	PCB66	440.09322
WELG04CP	97-190	Walleye Whole	PCB95	82.28819
WELG04CP	97-190	Walleye Whole	PCB87/115/81	61.76615
WELG04CP	97-190	Walleye Whole	PCB118	144.76877
WELG04CP	97-190	Walleye Whole	PCB153	131.48750
WELG04CP	97-190	Walleye Whole	PCB176	0.83231
WELG04CP	97-190	Walleye Whole	PCB169	1.35028

## SAMPLE RESULTS QUALIFIED AS A RESULT OF BLANK SPIKE AND SRM OUTLIERS (J/UJ-10)

Client_ID	Batch_ID	Matrix	Parameter	Concentration (ng/g, wet)
WELG05CP	97-190	Walleye Whole	PCB66	542.11292
WELG05CP	97-190	Walleye Whole	PCB95	92.25340
WELG05CP	97-190	Walleye Whole	PCB87/115/81	69.10568
WELG05CP	97-190	Walleye Whole	PCB118	173.22216
WELG05CP	97-190	Walleye Whole	PCB153	157.19023
WELG05CP	97-190	Walleye Whole	PCB176	2.16523
WELG05CP	97-190	Walleye Whole	PCB169	1.69577
WELG01CP	97-190	Walleye Whole	PCB66	369.64548
WELG01CP	97-190	Walleye Whole	PCB95	77.42129
WELG01CP	97-190	Walleye Whole	PCB87/115/81	62.33032
WELG01CP	97-190	Walleye Whole	PCB118	191.62172
WELG01CP	97-190	Walleye Whole	PCB153	203.62350
WELG01CP	97-190	Walleye Whole	PCB176	2.82216
WELG01CP	97-190	Walleye Whole	PCB169	2.39732
WELG02CP	97-190	Walleye Whole	PCB66	466.37483
WELG02CP	97-190	Walleye Whole	PCB95	95.49359
WELG02CP	97-190	Walleye Whole	PCB87/115/81	61.90176
WELG02CP	97-190	Walleye Whole	PCB118	174.36441
WELG02CP	97-190	Walleye Whole	PCB153	164.22295
WELG02CP	97-190	Walleye Whole	PCB169	1.55698
WELG03CP	97-190	Walleye Whole	PCB66	415.93033
WELG03CP	97-190	Walleye Whole	PCB95	100.32650
WELG03CP	97-190	Walleye Whole	PCB87/115/81	65.52732
WELG03CP	97-190	Walleye Whole	PCB118	187.99572
WELG03CP	97-190	Walleye Whole	PCB153	202.40298
WELG03CP	97-190	Walleye Whole	PCB176	1.90026
WELG03CP	97-190	Walleye Whole	PCB169	2.44463
BTEG01CP	97-191	B.Trout Whole	PCB95	32.74072
BTEG03CP	97-191	B.Trout Whole	PCB66	117.39217
BTEG03CP	97-191	B.Trout Whole	PCB95	30.90223
BTEG03CP	97-191	B.Trout Whole	PCB118	116.73981
BTEG03CP	97-191	B.Trout Whole	PCB153	158.43260
BTEG03CP	97-191	B.Trout Whole	PCB138/160/163	120.15413
BTEG03CP	97-191	B.Trout Whole	PCB180	42.04412
BTEG03CP	97-191	B.Trout Whole	PCB169	1.34341
BTEG04CP	97-191	B.Trout Whole	PCB95	24.87007
BTEG05CP	97-191	B.Trout Whole	PCB66	164.27513
BTEG05CP	97-191	B.Trout Whole	PCB95	35.42025
BTEG05CP	97-191	B.Trout Whole	PCB118	162.66380
BTEG05CP	97-191	B.Trout Whole	PCB153	211.58677
BTEG05CP	97-191	B.Trout Whole	PCB176	0.77516
BTEG05CP	97-191	B.Trout Whole	PCB138/160/163	160.67211
BTEG05CP	97-191	B.Trout Whole	PCB180	53.54520
BTEG05CP	97-191	B.Trout Whole	PCB169	1.27454
BTUG01CP	97-191	B.Trout Whole	PCB95	30.41482
BTUG02CP	97-191	B.Trout Whole	PCB95	23.76715
BTUG04CP	97-191	B.Trout Whole	PCB95	21.60728
BTUG05CP	97-191	B.Trout Whole	PCB95	27.97568
WEEG05CP	97-191	Walleye Whole	PCB66	672.56445
WEEG05CP	97-191	Walleye Whole	PCB95	133.67184

## SAMPLE RESULTS QUALIFIED AS A RESULT OF BLANK SPIKE AND SRM OUTLIERS (J/UJ-10)

Client_ID	Batch_ID	Matrix	Parameter	Concentration (ng/g, wet)
WEEG05CP	97-191	Walleye Whole	PCB118	324.45246
WEEG05CP	97-191	Walleye Whole	PCB153	464.42202
WEEG05CP	97-191	Walleye Whole	PCB176	4.40130
WEEG05CP	97-191	Walleye Whole	PCB138/160/163	472.40318
WEEG05CP	97-191	Walleye Whole	PCB180	137.25847
WEEG05CP	97-191	Walleye Whole	PCB169	5.17204
WEEG06CP	97-191	Walleye Whole	PCB66	593.83072
WEEG06CP	97-191	Walleye Whole	PCB95	158.17253
WEEG06CP	97-191	Walleye Whole	PCB118	296.22680
WEEG06CP	97-191	Walleye Whole	PCB153	407.54164
WEEG06CP	97-191	Walleye Whole	PCB176	4.43453
WEEG06CP	97-191	Walleye Whole	PCB138/160/163	417.57820
WEEG06CP	97-191	Walleye Whole	PCB180	124.09919
WEEG06CP	97-191	Walleye Whole	PCB169	4.61514
WEEG07CP	97-191	Walleye Whole	PCB66	1061.45469
WEEG07CP	97-191	Walleye Whole	PCB95	180.11058
WEEG07CP	97-191	Walleye Whole	PCB118	574.62261
WEEG07CP	97-191	Walleye Whole	PCB153	775.61102
WEEG07CP	97-191	Walleye Whole	PCB176	5.23457
WEEG07CP	97-191	Walleye Whole	PCB138/160/163	710.73864
WEEG07CP	97-191	Walleye Whole	PCB180	187.83725
WEEG07CP	97-191	Walleye Whole	PCB169	6.24752
WEEG08CP	97-191	Walleye Whole	PCB66	769.71423
WEEG08CP	97-191	Walleye Whole	PCB95	163.65783
WEEG08CP	97-191	Walleye Whole	PCB118	466.52899
WEEG08CP	97-191	Walleye Whole	PCB153	654.24995
WEEG08CP	97-191	Walleye Whole	PCB176	3.08746
WEEG08CP	97-191	Walleye Whole	PCB138/160/163	605.35534
WEEG08CP	97-191	Walleye Whole	PCB180	166.67238
WEEG08CP	97-191	Walleye Whole	PCB169	5.52434
WEEG10CP	97-191	Walleye Whole	PCB66	1770.43495
WEEG10CP	97-191	Walleye Whole	PCB95	298.03959
WEEG10CP	97-191	Walleye Whole	PCB118	983.16688
WEEG10CP	97-191	Walleye Whole	PCB153	1072.11776
WEEG10CP	97-191	Walleye Whole	PCB176	4.52897
WEEG10CP	97-191	Walleye Whole	PCB138/160/163	877.35862
WEEG10CP	97-191	Walleye Whole	PCB180	274.01203
WEEG10CP	97-191	Walleye Whole	PCB169	7.08917
WEEG11CP	97-191	Walleye Whole	PCB66	461.80057
WEEG11CP	97-191	Walleye Whole	PCB95	114.97922
WEEG11CP	97-191	Walleye Whole	PCB118	209.97651
WEEG11CP	97-191	Walleye Whole	PCB153	257.78468
WEEG11CP	97-191	Walleye Whole	PCB176	3.21162
WEEG11CP	97-191	Walleye Whole	PCB138/160/163	252.85243
WEEG11CP	97-191	Walleye Whole	PCB180	99.79975
WEEG11CP	97-191	Walleye Whole	PCB169	4.04364
WEUG01CP	97-191	Walleye Whole	PCB66	447.25055
WEUG01CP	97-191	Walleye Whole	PCB95	130.18832
WEUG01CP	97-191	Walleye Whole	PCB118	195.12208
WEUG01CP	97-191	Walleye Whole	PCB153	236.29354

## SAMPLE RESULTS QUALIFIED AS A RESULT OF BLANK SPIKE AND SRM OUTLIERS (J/UJ-10)

Client_ID	Batch_ID	Matrix	Parameter	Concentration (ng/g, wet)
WEUG01CP	97-191	Walleye Whole	PCB176	2.88719
WEUG01CP	97-191	Walleye Whole	PCB138/160/163	248.78818
WEUG01CP	97-191	Walleye Whole	PCB180	86.78450
WEUG01CP	97-191	Walleye Whole	PCB169	3.20138
WEUG03CP	97-191	Walleye Whole	PCB66	524.77593
WEUG03CP	97-191	Walleye Whole	PCB95	144.80335
WEUG03CP	97-191	Walleye Whole	PCB118	308.48427
WEUG03CP	97-191	Walleye Whole	PCB153	480.97790
WEUG03CP	97-191	Walleye Whole	PCB176	3.57025
WEUG03CP	97-191	Walleye Whole	PCB138/160/163	447.88667
WEUG03CP	97-191	Walleye Whole	PCB180	161.71550
WEUG03CP	97-191	Walleye Whole	PCB169	7.78640
BTEG02CP	97-192	B.Trout Whole	PCB118	95.87542
BTUG03CP	97-192	B.Trout Whole	PCB118	69.66250
EGLMF01WC-1	97-192	L.Trout Whole	PCB118	266.54243
EGLMF02WC-1	97-192	L.Trout Whole	PCB118	348.09430
EGLMF03WC-1	97-192	L.Trout Whole	PCB118	485.26712
EGLMF04WC-1	97-192	L.Trout Whole	PCB118	270.69644
EGLMF05WC-1	97-192	L.Trout Whole	PCB118	559.75962
EGLMF06WC-1	97-192	L.Trout Whole	PCB118	237.71889
EGLMF07WC-1	97-192	L.Trout Whole	PCB118	435.29363
EGLMF08WC-1	97-192	L.Trout Whole	PCB118	224.05929
EGLMF11WC-1	97-192	L.Trout Whole	PCB118	112.11703
EGLMF12WC-1	97-192	L.Trout Whole	PCB118	336.44410
WEEG02CP	97-192	Walleye Whole	PCB118	172.33138
WEEG09CP	97-192	Walleye Whole	PCB169	0.12292
WEEG09CP	97-192	Walleye Whole	PCB118	275.74328
WEFR07CP	97-192	Walleye Whole	PCB118	312.70665
WELG01CP	97-192	Walleye Whole	PCB118	251.21135
WELG06CP	97-192	Walleye Whole	PCB118	517.99117
WEUG02CP	97-192	Walleye Whole	PCB118	168.14965
WEWG04CP	97-192	Walleye Whole	PCB118	121.75901
96KICT03	97-306	Tern Eggs	PCB126	1.46916
96KICT03	97-306	Tern Eggs	PCB126	1.30666
96KICT10	97-306	Tern Eggs	PCB126	1.23320
96KICT10	97-306	Tern Eggs	PCB126	1.12070
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB66	50.37475
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB95	14.95517
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB95	7.24453
EGLMF07WC-1	97-306	L.Trout Whole	PCB126	1.37184
EGLMF07WC-1	97-306	L.Trout Whole	PCB126	1.96529
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB66	34.23498
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB95	9.31798
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB95	5.53896
EGLMF09WC-1	97-306	L.Trout Whole	PCB66	303.94919
EGLMF09WC-1	97-306	L.Trout Whole	PCB95	83.95568
EGLMF09WC-1	97-306	L.Trout Whole	PCB66	471.90089
EGLMF09WC-1	97-306	L.Trout Whole	PCB95	117.61421
EGLMF09WC-1	97-306	L.Trout Whole	PCB126	2.17774
EGLMF09WC-1	97-306	L.Trout Whole	PCB126	1.72864

## SAMPLE RESULTS QUALIFIED AS A RESULT OF BLANK SPIKE AND SRM OUTLIERS (J/UJ-10)

Client_ID	Batch_ID	Matrix	Parameter	Concentration (ng/g, wet)
EGLMF10WC-1	97-306	L.Trout Whole	PCB66	168.86992
EGLMF10WC-1	97-306	L.Trout Whole	PCB95	44.75489
EGLMF10WC-1	97-306	L.Trout Whole	PCB126	0.20755
EGLMF11WC-1	97-306	L.Trout Whole	PCB126	0.68170
TEKIB48	97-306	Tern Eggs	PCB126	0.80491
WEWG04CP	97-306	Walleye Whole	PCB126	0.93064
WEWG04CP	97-306	Walleye Whole	PCB126	0.70704
BTUG03CP	97-312	B.Trout Whole	PCB37	0.27924
BTUG03CP	97-312	B.Trout Whole	PCB37	0.39659
EGLMF01WC-1	97-312	L.Trout Whole	PCB37	0.25142
EGLMF02WC-1	97-312	L.Trout Whole	PCB37	0.38638
EGLMF03WC-1	97-312	L.Trout Whole	PCB37	0.20341
EGLMF03WC-1	97-312	L.Trout Whole	PCB37	0.23220
EGLMF04WC-1	97-312	L.Trout Whole	PCB37	0.30943
EGLMF05WC-1	97-312	L.Trout Whole	PCB37	0.43653
EGLMF08WC-1	97-312	L.Trout Whole	PCB37	0.00000
EGLMF12WC-1	97-312	L.Trout Whole	PCB37	0.19078

## SAMPLE RESULTS QUALIFIED AS A RESULT OF DUPLICATE RPD OUTLIERS (J/UJ-9)

Client ID	Batch ID	Matrix	Parameter	Concentration (ng/g, wet)
96KICT01	97-126	Tern Eggs	PCB37	0.72666
96KICT01	97-126	Tern Eggs	PCB81	0.99183
96KICT01	97-126	Tern Eggs	PCB63	50.82982
96KICT01	97-126	Tern Eggs	PCB132	141.44357
96KICT03	97-126	Tern Eggs	PCB63	26.37473
96KICT03	97-126	Tern Eggs	PCB132	32.68346
96KICT05	97-126	Tern Eggs	PCB132	29.96365
96KICT05	97-126	Tern Eggs	PCB63	48.25779
96KICT07	97-126	Tern Eggs	PCB63	7.18822
96KICT07	97-126	Tern Eggs	PCB132	54.35061
96KICT09	97-126	Tern Eggs	PCB132	39.62165
96KICT09	97-126	Tern Eggs	PCB63	41.85027
96KICT10	97-126	Tern Eggs	PCB63	29.19699
96KICT10	97-126	Tern Eggs	PCB132	269.00683
TEKIB06	97-126	Tern Eggs	PCB37	0.18066
TEKIB06	97-126	Tern Eggs	PCB81	1.22174
TEKIB06	97-126	Tern Eggs	PCB63	26.17184
TEKIB06	97-126	Tern Eggs	PCB132	42.18158
TEKIB18	97-126	Tern Eggs	PCB132	32.27604
TEKIB18	97-126	Tern Eggs	PCB63	38.92911
TEKIB24	97-126	Tern Eggs	PCB37	0.20807
TEKIB24	97-126	Tern Eggs	PCB81	0.71252
TEKIB24	97-126	Tern Eggs	PCB63	21.17520
TEKIB24	97-126	Tern Eggs	PCB132	54.53029
TEKIB30	97-126	Tern Eggs	PCB37	0.17747
TEKIB30	97-126	Tern Eggs	PCB81	0.51214
TEKIB30	97-126	Tern Eggs	PCB63	17.85565
TEKIB30	97-126	Tern Eggs	PCB132	42.98764
TEKIB48	97-126	Tern Eggs	PCB63	31.78046
TEKIB48	97-126	Tern Eggs	PCB132	52.20509
TEKIB60	97-126	Tern Eggs	PCB37	0.28751
TEKIB60	97-126	Tern Eggs	PCB81	0.63917
TEKIB60	97-126	Tern Eggs	PCB63	30.24157
TEKIB60	97-126	Tern Eggs	PCB132	81.48316
EGLMF01FC-1	97-129	Lake Trout Eggs	PCB77	0.11498
EGLMF01FC-1	97-129	Lake Trout Eggs	PCB126	0.05915
EGLMF02FC-1	97-129	Lake Trout Eggs	PCB77	1.31113
EGLMF02FC-1	97-129	Lake Trout Eggs	PCB126	0.31115
EGLMF03FC-1	97-129	Lake Trout Eggs	PCB77	0.52903
EGLMF03FC-1	97-129	Lake Trout Eggs	PCB126	0.17619
EGLMF04FC-1	97-129	Lake Trout Eggs	PCB77	0.85347
EGLMF04FC-1	97-129	Lake Trout Eggs	PCB126	0.19730
EGLMF05FC-1	97-129	Lake Trout Eggs	PCB77	1.22824
EGLMF05FC-1	97-129	Lake Trout Eggs	PCB126	0.31402
EGLMF06FC-1	97-129	Lake Trout Eggs	PCB77	1.13212
EGLMF06FC-1	97-129	Lake Trout Eggs	PCB126	0.29061
EGLMF07FC-1	97-129	Lake Trout Eggs	PCB77	1.36861
EGLMF07FC-1	97-129	Lake Trout Eggs	PCB126	0.32697
EGLMF08FC-1	97-129	Lake Trout Eggs	PCB77	0.21401

## SAMPLE RESULTS QUALIFIED AS A RESULT OF DUPLICATE RPD OUTLIERS (J/UJ-9)

Client ID	Batch ID	Matrix	Parameter	Concentration (ng/g, wet)
EGLMF09FC-1	97-129	Lake Trout Eggs	PCB77	1.25099
EGLMF09FC-1	97-129	Lake Trout Eggs	PCB126	0.34722
EGLMF10FC-1	97-129	Lake Trout Eggs	PCB77	2.09536
EGLMF10FC-1	97-129	Lake Trout Eggs	PCB126	0.42840
EGLMF11FC-1	97-129	Lake Trout Eggs	PCB77	0.65290
EGLMF11FC-1	97-129	Lake Trout Eggs	PCB126	0.07803
EGLMF12FC-1	97-129	Lake Trout Eggs	PCB77	0.15594
EGLMF12FC-1	97-129	Lake Trout Eggs	PCB126	0.18373
BTEG01CP	97-191	B.Trout Whole	PCB85	309.21389
BTEG03CP	97-191	B.Trout Whole	PCB85	402.69191
BTEG04CP	97-191	B.Trout Whole	PCB85	326.33964
BTEG05CP	97-191	B.Trout Whole	PCB85	494.54958
BTUG01CP	97-191	B.Trout Whole	PCB85	378.32038
BTUG02CP	97-191	B.Trout Whole	PCB85	327.73007
BTUG04CP	97-191	B.Trout Whole	PCB85	328.96003
BTUG05CP	97-191	B.Trout Whole	PCB85	342.65271
WEEG05CP	97-191	Walleye Whole	PCB85	2162.94313
WEEG06CP	97-191	Walleye Whole	PCB85	1943.59612
WEEG07CP	97-191	Walleye Whole	PCB85	2167.03630
WEEG08CP	97-191	Walleye Whole	PCB85	2133.26683
WEEG10CP	97-191	Walleye Whole	PCB85	2205.65340
WEEG11CP	97-191	Walleye Whole	PCB85	1382.98217
WEUG01CP	97-191	Walleye Whole	PCB85	1344.32135
WEUG03CP	97-191	Walleye Whole	PCB85	2364.90738
BTEG02CP	97-192	B.Trout Whole	PCB85	781.81935
BTEG02CP	97-192	B.Trout Whole	PCB180	41.81165
BTUG03CP	97-192	B.Trout Whole	PCB85	285.18549
BTUG03CP	97-192	B.Trout Whole	PCB180	23.08871
EGLMF01WC-1	97-192	L.Trout Whole	PCB85	2613.14572
EGLMF01WC-1	97-192	L.Trout Whole	PCB180	127.56807
EGLMF02WC-1	97-192	L.Trout Whole	PCB85	2701.26525
EGLMF02WC-1	97-192	L.Trout Whole	PCB180	159.63264
EGLMF03WC-1	97-192	L.Trout Whole	PCB85	3452.65478
EGLMF03WC-1	97-192	L.Trout Whole	PCB180	221.47521
EGLMF04WC-1	97-192	L.Trout Whole	PCB85	2614.91961
EGLMF04WC-1	97-192	L.Trout Whole	PCB180	133.07266
EGLMF05WC-1	97-192	L.Trout Whole	PCB85	2079.80254
EGLMF05WC-1	97-192	L.Trout Whole	PCB180	226.42978
EGLMF06WC-1	97-192	L.Trout Whole	PCB85	2629.62183
EGLMF06WC-1	97-192	L.Trout Whole	PCB180	129.35301
EGLMF07WC-1	97-192	L.Trout Whole	PCB85	2129.52628
EGLMF07WC-1	97-192	L.Trout Whole	PCB180	179.67450
EGLMF08WC-1	97-192	L.Trout Whole	PCB85	2508.76791
EGLMF08WC-1	97-192	L.Trout Whole	PCB180	119.99790
EGLMF11WC-1	97-192	L.Trout Whole	PCB85	1213.53804
EGLMF11WC-1	97-192	L.Trout Whole	PCB180	59.45960
EGLMF12WC-1	97-192	L.Trout Whole	PCB85	2141.05995
EGLMF12WC-1	97-192	L.Trout Whole	PCB180	140.56961
WEEG02CP	97-192	Walleye Whole	PCB85	947.90876

## SAMPLE RESULTS QUALIFIED AS A RESULT OF DUPLICATE RPD OUTLIERS (J/UJ-9)

Client ID	Batch ID	Matrix	Parameter	Concentration (ng/g, wet)
WEEG02CP	97-192	Walleye Whole	PCB180	62.99027
WEEG09CP	97-192	Walleye Whole	PCB85	1987.90089
WEEG09CP	97-192	Walleye Whole	PCB180	131.83004
WEFR07CP	97-192	Walleye Whole	PCB85	1123.27999
WEFR07CP	97-192	Walleye Whole	PCB180	37.85564
WELG01CP	97-192	Walleye Whole	PCB85	1297.49659
WELG01CP	97-192	Walleye Whole	PCB180	104.00942
WELG06CP	97-192	Walleye Whole	PCB85	1619.34567
WELG06CP	97-192	Walleye Whole	PCB180	155.97690
WEUG02CP	97-192	Walleye Whole	PCB85	1243.30206
WEUG02CP	97-192	Walleye Whole	PCB180	82.86603
WEWG04CP	97-192	Walleye Whole	PCB85	399.66711
WEWG04CP	97-192	Walleye Whole	PCB180	43.91320
96KICT05	97-274	Tern Eggs	PCB126	1.26048
96KICT07	97-274	Tern Eggs	PCB126	0.81603
96KICT09	97-274	Tern Eggs	PCB126	0.92498
BTEG02CP	97-274	B.Trout Whole	PCB126	0.54150
EGLMF06WC-1	97-274	L.Trout Whole	PCB126	0.29679
TEKIB18	97-274	Tern Eggs	PCB126	0.67831
WEFR07CP	97-274	Walleye Whole	PCB126	0.86155
WEFR07CP	97-274	Walleye Whole	PCB126	0.47479
WELG06CP	97-274	Walleye Whole	PCB126	1.14246
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB31	9.64066
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB28	13.92704
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB52	25.58797
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB49	18.19334
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB47/75	13.86909
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB44	17.32018
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB42/37	4.51004
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB63	5.29990
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB74	19.84732
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB70/76	36.24314
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB66	50.37475
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB95	14.95517
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB91	8.18757
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB56/60	17.17525
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB92	11.21620
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB84	17.93270
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB101/90	41.16918
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB99	38.50616
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB83	5.11670
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB97	13.16928
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB87/115/81	15.33698
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB85	21.88658
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB110/77	46.25477
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB82	6.37565
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB151	6.34463
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB135/144	8.97465
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB107/147	10.85050

## SAMPLE RESULTS QUALIFIED AS A RESULT OF DUPLICATE RPD OUTLIERS (J/UJ-9)

Client ID	Batch ID	Matrix	Parameter	Concentration (ng/g, wet)
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB149/123	28.73250
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB118	60.28390
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB131	8.47346
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB146	14.31541
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB153	73.38141
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB132	7.82117
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB105	28.44523
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB141/179	6.94632
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB176	7.56233
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB130	5.42227
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB138/160/163	76.54443
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB178	4.33121
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB187/182	19.25765
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB183	7.29970
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB128	13.23936
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB174	6.91948
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB177	6.37137
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB171/202	5.98787
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB156	7.94473
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB180	41.70070
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB170/190	9.80318
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB199	6.40219
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB203/196	6.10408
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB31	5.29588
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB28	7.55210
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB52	12.73544
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB49	9.05152
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB47/75	7.18207
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB44	8.07298
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB42/37	2.13729
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB63	3.52492
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB74	9.97517
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB70/76	17.39217
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB66	25.18594
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB95	7.24453
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB91	3.72247
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB56/60	9.00404
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB92	4.48948
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB84	8.53712
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB101/90	20.13333
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB99	18.71490
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB83	2.82290
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB97	6.03594
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB87/115/81	7.67239
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB85	6.56886
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB110/77	21.50539
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB82	3.05387
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB151	2.93704
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB135/144	4.14697

## SAMPLE RESULTS QUALIFIED AS A RESULT OF DUPLICATE RPD OUTLIERS (J/UJ-9)

Client ID	Batch ID	Matrix	Parameter	Concentration (ng/g, wet)
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB107/147	4.98443
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB149/123	13.07534
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB118	27.92534
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB131	3.82946
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB146	6.65185
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB153	33.04066
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB132	3.44924
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB105	13.47929
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB141/179	3.04764
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB176	3.35918
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB130	2.48316
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB138/160/163	30.51557
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB178	1.68678
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB187/182	8.83485
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB183	3.15067
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB128	6.12500
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB174	2.94966
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB177	2.83081
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB171/202	2.51793
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB156	3.37980
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB180	18.11751
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB170/190	4.78763
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB199	2.88855
EGLMF01FC-1	97-306	Lake Trout Eggs	PCB203/196	2.71288
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB31	6.31117
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB28	9.36571
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB52	16.81621
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB49	12.26067
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB47/75	9.15484
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB44	10.98063
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB74	13.68577
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB70/76	23.31798
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB66	34.23498
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB95	9.31798
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB91	5.67490
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB56/60	11.04219
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB92	7.28528
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB84	10.80069
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB101/90	29.82540
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB99	24.86206
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB97	8.34002
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB87/115/81	10.24881
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB85	7.25395
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB110/77	28.51937
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB135/144	5.69289
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB107/147	6.10899
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB149/123	18.93913
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB118	37.14209
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB131	4.41097

## SAMPLE RESULTS QUALIFIED AS A RESULT OF DUPLICATE RPD OUTLIERS (J/UJ-9)

Client ID	Batch ID	Matrix	Parameter	Concentration (ng/g, wet)
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB146	8.78261
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB153	43.08300
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB132	5.67095
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB105	17.04239
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB138/160/163	44.95573
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB187/182	12.71532
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB183	4.40287
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB128	8.26789
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB174	4.42233
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB177	4.05929
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB180	26.67698
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB170/190	7.64634
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB199	4.23775
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB203/196	3.97885
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB31	3.30519
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB28	4.64253
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB52	8.96023
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB49	6.77687
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB47/75	4.97825
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB44	5.48896
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB74	7.17638
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB70/76	12.02151
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB66	17.12695
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB91	3.05990
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB56/60	5.77021
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB92	4.01769
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB84	5.56916
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB101/90	15.76696
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB99	13.29935
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB97	4.48255
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB87/115/81	5.91721
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB85	4.08101
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB110/77	15.59464
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB135/144	2.91721
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB107/147	3.50430
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB149/123	10.47938
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB118	19.71631
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB131	2.62037
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB146	4.80787
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB153	24.76250
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB132	3.01015
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB105	9.38166
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB138/160/163	25.58620
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB187/182	7.15357
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB183	2.56356
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB128	4.38571
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB174	2.52370
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB177	2.23742
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB180	14.23953

## SAMPLE RESULTS QUALIFIED AS A RESULT OF DUPLICATE RPD OUTLIERS (J/UJ-9)

Client ID	Batch ID	Matrix	Parameter	Concentration (ng/g, wet)
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB170/190	4.03807
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB199	2.44245
EGLMF08FC-1	97-306	Lake Trout Eggs	PCB203/196	2.32817
EGLMF10WC-1	97-306	L.Trout Whole	PCB31	31.75677
EGLMF10WC-1	97-306	L.Trout Whole	PCB28	43.99925
EGLMF10WC-1	97-306	L.Trout Whole	PCB52	87.26316
EGLMF10WC-1	97-306	L.Trout Whole	PCB49	54.65789
EGLMF10WC-1	97-306	L.Trout Whole	PCB47/75	50.54023
EGLMF10WC-1	97-306	L.Trout Whole	PCB44	45.83421
EGLMF10WC-1	97-306	L.Trout Whole	PCB63	28.05564
EGLMF10WC-1	97-306	L.Trout Whole	PCB74	76.95602
EGLMF10WC-1	97-306	L.Trout Whole	PCB70/76	115.54286
EGLMF10WC-1	97-306	L.Trout Whole	PCB66	168.86992
EGLMF10WC-1	97-306	L.Trout Whole	PCB95	44.75489
EGLMF10WC-1	97-306	L.Trout Whole	PCB91	26.90789
EGLMF10WC-1	97-306	L.Trout Whole	PCB56/60	49.20150
EGLMF10WC-1	97-306	L.Trout Whole	PCB92	26.76278
EGLMF10WC-1	97-306	L.Trout Whole	PCB84	59.24286
EGLMF10WC-1	97-306	L.Trout Whole	PCB101/90	154.70038
EGLMF10WC-1	97-306	L.Trout Whole	PCB99	135.20865
EGLMF10WC-1	97-306	L.Trout Whole	PCB97	49.66316
EGLMF10WC-1	97-306	L.Trout Whole	PCB87/115/81	56.95226
EGLMF10WC-1	97-306	L.Trout Whole	PCB85	50.13872
EGLMF10WC-1	97-306	L.Trout Whole	PCB110/77	130.58835
EGLMF10WC-1	97-306	L.Trout Whole	PCB82	21.43308
EGLMF10WC-1	97-306	L.Trout Whole	PCB151	25.89023
EGLMF10WC-1	97-306	L.Trout Whole	PCB135/144	31.79173
EGLMF10WC-1	97-306	L.Trout Whole	PCB107/147	35.38008
EGLMF10WC-1	97-306	L.Trout Whole	PCB149/123	108.41015
EGLMF10WC-1	97-306	L.Trout Whole	PCB118	204.25977
EGLMF10WC-1	97-306	L.Trout Whole	PCB131	20.74774
EGLMF10WC-1	97-306	L.Trout Whole	PCB146	53.16015
EGLMF10WC-1	97-306	L.Trout Whole	PCB153	270.65526
EGLMF10WC-1	97-306	L.Trout Whole	PCB132	37.74962
EGLMF10WC-1	97-306	L.Trout Whole	PCB105	87.66429
EGLMF10WC-1	97-306	L.Trout Whole	PCB141/179	21.55301
EGLMF10WC-1	97-306	L.Trout Whole	PCB176	23.41654
EGLMF10WC-1	97-306	L.Trout Whole	PCB130	18.09248
EGLMF10WC-1	97-306	L.Trout Whole	PCB138/160/163	228.50902
EGLMF10WC-1	97-306	L.Trout Whole	PCB178	19.40902
EGLMF10WC-1	97-306	L.Trout Whole	PCB187/182	86.18722
EGLMF10WC-1	97-306	L.Trout Whole	PCB183	33.90677
EGLMF10WC-1	97-306	L.Trout Whole	PCB128	43.90038
EGLMF10WC-1	97-306	L.Trout Whole	PCB174	29.28872
EGLMF10WC-1	97-306	L.Trout Whole	PCB177	26.36015
EGLMF10WC-1	97-306	L.Trout Whole	PCB171/202	20.88120
EGLMF10WC-1	97-306	L.Trout Whole	PCB156	28.97030
EGLMF10WC-1	97-306	L.Trout Whole	PCB180	152.99774
EGLMF10WC-1	97-306	L.Trout Whole	PCB170/190	41.72707

**SAMPLE RESULTS QUALIFIED AS A RESULT OF DUPLICATE RPD OUTLIERS (J/UJ-9)**

<b>Client ID</b>	<b>Batch ID</b>	<b>Matrix</b>	<b>Parameter</b>	<b>Concentration (ng/g, wet)</b>
EGLMF10WC-1	97-306	L.Trout Whole	PCB199	26.92293
EGLMF10WC-1	97-306	L.Trout Whole	PCB203/196	28.33609



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**APPENDIX C**  
**PROCEDURE AND RESULTS OF WATERFOWL COLLECTION BY USFWS**  
**IN THE ASSESSMENT AREA, 1997**

**Standard Operating Procedure for Collection, Preparation, Transport and Storage of Samples**

**Report by Dr. T. Custer et al. to USFWS, Green Bay Office**

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# STANDARD OPERATING PROCEDURE FOR THE COLLECTION, PREPARATION, TRANSPORT, AND STORAGE OF WATERFOWL CARCASSES FROM GREEN BAY, WISCONSIN

## 1. INTRODUCTION AND STUDY OBJECTIVES

This Standard Operating Procedure (SOP) contains the objectives, methods, and approaches for the collection, preparation, transport, and storage of waterfowl carcasses to be collected from Green Bay, Wisconsin, for the Fox River/Green Bay Natural Resource Damage Assessment (NRDA). Waterfowl tissues will be analyzed for contaminants by an analytical laboratory. A subsequent SOP will describe the laboratory analytical methods that will be employed.

The objective of the study is to:

- ▶ determine organochlorine concentrations in carcasses and breast muscle tissue of waterfowl breeding and wintering in Green Bay and the lower Fox River, Wisconsin.

Adult waterfowl will be collected during two periods in the winter of 1997/1988 (September/October and October/November), and during the 1988 nesting season and will be analyzed for organochlorines, including PCBs. The field team leader for the collections will be Dr. Thomas Custer (U.S. Geological Survey, Upper Mississippi Science Center, LaCrosse, WI).

## 2. FIELD PROCEDURES

### 2.1 WATERFOWL COLLECTION LOCATIONS

During the winter of 1997, waterfowl distribution and abundance will be measured through aerial surveys of the Fox River and Green Bay by Wisconsin DNR. Local hunters may also assist in identifying suitable areas to collect waterfowl.

### 2.2 WATERFOWL COLLECTION

A variety of collection methods may be employed. These include but may not be limited to 1) shooting from a fast-moving boat, 2) shooting from a skull boat, 3) jump shooting birds from

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shore, 4) or hunting from a blind or lay-out boat. The team leader or his representative will be present during all collections.

The species and numbers of birds that will be collected are shown in Table 1.

<b>Table 1</b>						
<b>Proposed waterfowl sampling effort for winter 1997</b>						
Numbers actually collected may be lower and/or species distributions may change, depending on availability of birds.						
Species	Number to be Collected		Number to be Analyzed			
	Sept/Oct	Oct/Nov	Sept/Oct		Oct/Nov	
			Carcass*	Breast	Carcass*	Breast
Lesser Scaup	10	10	3	10	3	10
Common Goldeneye	0	10	0	0	3	10
Red-breasted Merganser**	0	10	0	0	3	10
Mallard**	5	0	3	5	0	0
* Carcass samples will be randomly selected from among the total sample. ** Mallards and mergansers (10 each) will also be collected in the summers of 1997 and 1998. Summary: Maximum of 80 samples for OC analyses						

On collection, each bird will be given a unique numerical identifier in the field. This number will be written on a tag and the tag tied to one leg. All identification numbers will be recorded in the field logbook. The identification system for waterfowl samples collected for contaminant analyses consists of the following code:

**WF-XX-YY-00**

where:

- ▶ **WF** is a two-letter code designating the waterfowl collection effort.
- ▶ **XX** is a unique two-letter code designating the collection location
- ▶ **YY** is a waterfowl species identifier (e.g.: LS = lesser scaup, etc)
- ▶ **00** is a unique two-number code designating the number assigned to this individual. Waterfowl will be numbered starting at "01."

Once uniquely identified, each bird will be placed in separate self-sealing plastic bags for transport to the USFWS Field Office in Green Bay.

### **2.3 FIELD DOCUMENTATION**

The field team will document its sampling activities and field measurements in a dedicated, paginated, bound field logbook. Sampling locations will be clearly identified on photocopies of appropriate topographical maps and described in the field notebook. Entries in the field notebook and map marking will be done with waterproof ink, and corrections will be made with a single line through the error accompanied by the correction date and corrector's initials. The field team leader will be responsible for maintenance and proper archiving of these field notebooks.

The following information will be recorded in the field logbooks:

- ▶ site and project name
- ▶ each sampler's name and professional affiliation
- ▶ date and time of collection, field activity, or field measurement
- ▶ exact location of collection
- ▶ method of collection
- ▶ identification numbers of samples collected
- ▶ number and type of samples collected
- ▶ any difficulties encountered or necessary deviations from this SOP
- ▶ any other pertinent field observations.

Maps will be marked with a sampling location code, e.g., KI for Kidney Island, written within a circle. The field notebook page number corresponding to each sampling location will be marked adjacent to the sampling location circle.

Upon completion of each day's field activities, the notes will be reviewed by the field recorder and sampler and any necessary corrections made. The field recorder will sign and date each page.

### **2.4 PROCESSING AND STORAGE OF WATERFOWL TISSUES**

The field team leader or a designated representative will transport the waterfowl to the USFWS Field Office in Green Bay. Immediately on returning from the field to the laboratory, the birds will be weighed and wing length measured. Measurements will be made using an electronic balance and a ruler and will include:

- ▶ wing length (to the closest 1.0 mm).
  - ▶ weight (to the closest 0.1g).
-

- ▶ sex.
- ▶ age.

These measurements will be recorded in the field notebook.

After the above measurements are taken, the birds will be plucked, the contents of the esophagus, proventriculus and gizzard removed. The right side of the breast and associated skin will then be surgically removed.

After each dissection, the surgical equipment and the cutting board or table surface on which the dissections take place will be decontaminated according to the following procedure:

- ▶ pre-wash, using deionized water and scrub brush as necessary
- ▶ rinse thoroughly with ultra-clean acetone
- ▶ rinse thoroughly with ultra-clean hexane
- ▶ rinse again with ultra-clean acetone
- ▶ rinse thoroughly three times with deionized water.

The breast and associated skin will be weighed and wrapped in aluminum foil and sealed in an individual plastic bag. The remainder of the carcass will be weighed and wrapped in aluminum foil and sealed in an individual plastic bag. The letter 'M' for muscle or a 'C' for carcass (see below) will be attached to the labels as appropriate. The samples will be stored in a freezer before shipment to the analytical laboratory. The final identification system for waterfowl samples collected for contaminant analyses consists of the following code:

**WF-XX-YY-00-T**

where:

- ▶ T is a one-letter code designating the waterfowl tissue (C = carcass, M = breast muscle)

## **2.5 CHAIN OF CUSTODY**

The chain of custody will start when waterfowl are collected. Each bird will be given a unique numerical identifier in the field. This number will be written on a tag and the tag attached to the carcass. Once identified in this way, the waterfowl collected during each sampling event will be placed (each sample within its own self-sealing plastic bag) in a communal container under the custody of Dr. Tom Custer or a designated stand-in. Each of the self-sealing plastic bags will be labeled with the appropriate sample identifier. The bags will be stored frozen in one or more shipping containers which will be sealed with custody seals (to detect unauthorized tampering

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with samples after sample collection until the time of use or analysis), and contain chain of custody forms with the following information, as appropriate:

- ▶ project name
- ▶ waterfowl identifiers (unique for each sample)
- ▶ name and signature of field recorder
- ▶ date and time of beginning of sample collection
- ▶ chain of custody seal number
- ▶ signatures of persons involved in the chain of possession
- ▶ inclusive dates and times of possession
- ▶ method and date of sample shipment.

At the appropriate time, the entire sealed container(s) will be shipped to the analytical laboratory.

The designated field sample eustodian will be personally responsible for the care and custody of the samples until they are transferred or properly dispatched. A sample is in the custody of an individual if any of the following occur:

- ▶ The sample is in the individual's possession.
- ▶ The sample is within view after being in possession.
- ▶ The sample is in a locked or sealed container that prevents tampering after being in possession.
- ▶ The sample is in a designated secure area.

Every transfer of custody will be noted with the date and time of transfer and signed for on the chain of custody record. The number of custody transfers will be kept to a minimum.

## **2.7 FIELD EQUIPMENT**

The following list of equipment will be required in the field:

- ▶ SOPs (one copy for each team member)
  - ▶ waders/hip boots (all crew members)
  - ▶ field log books
  - ▶ marking pens and pencils
  - ▶ labels and labeling tape
  - ▶ string
  - ▶ self-sealing plastic bags
  - ▶ chain of custody forms and seals
  - ▶ shotguns and shells (steel shot)
-

## **2.8 DEVIATIONS FROM THIS SOP**

If field conditions necessitate any deviations from this SOP the Field Team Leader will document them in the field note book and in an addendum to this SOP.

**Concentrations of polychlorinated biphenyls in tissues of waterfowl  
from Green Bay, Wisconsin and nearby Lake Michigan**

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## Introduction

Green Bay is contaminated with polychlorinated biphenyls (PCBs), most of which reportedly originated from the deinking and repulping of carbonless paper at paper mills on the Fox River (Fig. 1) (Sullivan *et al.* 1983). Elevated PCB concentrations have been documented in Green Bay sediment (Sullivan *et al.* 1983, Hermanson *et al.* 1991, Ankley *et al.* 1992, Velleux and Endicot 1994, Manchester-Neesvig *et al.* 1996), fish (Sullivan *et al.* 1983), and birds (Ankley *et al.* 1993, Custer and Custer 1995, Harris *et al.* 1993, Rattner *et al.* 1993, Hoffman *et al.* 1993, Kubiak *et al.* 1989, Custer *et al.* 1998, Custer *et al.* 1999). The Wisconsin Department of Natural Resources (WDNR) has issued a consumption advisory on mallards (*Anas platyrhynchos*) obtained from Green Bay, Wisconsin because of high levels of PCBs in their tissues.

Zebra mussels (*Dreissena polymorpha*) have reached high densities in the Great Lakes, including Green Bay, since their introduction in the mid 1980s. Densities of zebra mussels over 700,000/m<sup>2</sup> have been reported at power plants on Lake Erie (Kovalak *et al.* 1993) and as many as 342,000/m<sup>2</sup> on fish-spawning reefs in Lake Erie (Leach 1993). Zebra mussel biomass can be as high as 3.6 kg/m<sup>2</sup> (Custer and Custer 1997). The bioaccumulation capacities of zebra mussels (Brieger and Hunter 1993, Busch and Schuchardt 1991, Mersch *et al.* 1992) may enhance the transfer of contaminants to waterfowl (de Kock and Bowmer 1993). Contaminants, if high enough, can negatively affect waterfowl reproduction (de Kock and Bowmer 1993) or may have secondary effects as a contaminant source for Bald Eagles (*Haliaeetus leucocephalus*), other raptors, and humans.

Waterfowl are now migrating through and wintering in parts of the Great Lakes in larger numbers than they had immediately prior to the zebra mussel invasion (Wormington and Leach 1992). This increase has probably been due to the presence of zebra mussels, a now abundant and easily captured food source.

Zebra mussels are the primary food now for lesser scaup (*Aythya affinis*) and common goldeneye (*Bucephala clangula*) in the Great Lakes, especially in western Lake Erie (Custer and Custer 1996, Hamilton *et al.* 1994). Ninety-eight percent of lesser scaup diet, 79% of common goldeneye diet, 24% of bufflehead (*Bucephala albeola*) diet, but < 10% of canvasback (*Aythya valisineria*) diet are now zebra mussels (Custer and Custer 1996). The consequences of this food shift are mostly unknown, however, the potential for contaminant transfer may be high. The Great Lakes are an area of known contamination (Government of Canada 1991). Diving ducks collected in the Detroit River in 1980 had high organochlorine concentrations (Smith *et al.* 1985). Chlorinated hydrocarbon contaminants were still present in waterfowl from the Detroit River in the early 1990s (Mazak *et al.* 1997).

Human consumption advisory levels for PCB concentrations in edible poultry are available for Canada (0.5  $\mu\text{g/g}$  lipid weight, Health and Welfare Canada 1991) and the United States (3.0  $\mu\text{g/g}$  lipid weight, FDA 1979). Furthermore, PCB concentrations can be compared to the 'do not eat' category (1.9  $\mu\text{g/g}$  wet weight) under proposed guidelines for a uniform Great Lakes sport fish consumption advisory (Anderson *et al.* 1993).

The objective of the study was to determine whether PCB concentrations in tissues of waterfowl breeding and wintering in Green Bay, Wisconsin exceeded human consumption advisory levels.

## Methods

Waterfowl were collected by shotgun using steel shot in Green Bay and Lake Michigan during June to November 1997 under appropriate state and federal collecting permits. After collection, the birds were weighed (0.1 g) in the laboratory and in the case of lesser and greater scaup the wing length (1.0 mm) was measured. The breast of the birds was plucked and the right side of the breast and associated skin were then surgically removed. The breast and associated skin were individually weighed, wrapped in aluminum foil, sealed in an individual plastic bag, and frozen at -20 °C. Age and sex of waterfowl was determined using plumage and cloacal characteristics (Carney 1964). The remainder of the carcass was weighed, wrapped in aluminum foil, sealed in an individual plastic bag, and frozen at -20 °C.

The following organochlorines were analyzed in waterfowl muscle and skin samples by Mississippi State Chemical Laboratory, Mississippi State, Mississippi, USA:  $\alpha$ -,  $\beta$ -,  $\gamma$ - and  $\delta$ -hexachlorocyclohexane (HCH);  $\alpha$ - and  $\delta$ - chlordane; oxychlordane; *cis*-nonachlor; *trans*-nonachlor; dieldrin; endrin; hexachlorobenzene (HCB); heptachlor epoxide; mirex; toxaphene; *o,p'*-dichlorodiphenyldichloroethane(DDD); *o,p'*-dichlorodiphenyldichloroethylene (DDE); *o,p'*-dichlorodiphenyltrichloroethane (DDT); *p,p'*-DDD; *p,p'*-DDE; *p,p'*-DDT; and total PCBs. Samples were homogenized, mixed with sodium sulfate and soxhlet extracted with hexane. After the lipid determination, lipids were removed by florisil column chromatography. Following silicic acid column chromatography, pesticides and total PCBs were determined by electron capture gas chromatography. Total PCBs were estimated based on Aroclor equivalents. The nominal limit of detection for organochlorines 0.01  $\mu\text{g/g}$  wet weight, except for mallards which was 0.02  $\mu\text{g/g}$ . The number of spikes, duplicates and blanks was 10% of the total number

of samples analyzed. Concentrations were not adjusted for recovery which averaged 90% for all organochlorines. Organochlorine concentrations in breast muscle without skin, skin associated with the breast muscle, and breast muscle with skin are reported on a wet weight and lipid weight basis. Breast muscle from all waterfowl collected were analyzed for organochlorines. Because of budgetary constraints, not all the skins associated with breast muscle were analyzed for organochlorines.

### Results and Discussion

Waterfowl were collected from three locations in Green Bay and Lake Michigan in 1997 (Fig. 1). For mallards collected in June (n=10, Tables 1 and 2) breast muscle of 5, 4, and 0 birds were above the Canadian PCB consumption advisory (0.5 µg/g lipid weight, Health and Welfare Canada 1991), United States PCB consumption advisory (3.0 µg/g lipid weight, FDA 1979), and the Great Lakes sport fish consumption advisory (1.9 µg/g wet weight, Anderson *et al.* 1993), respectively. When skin was added to the muscle, all 10 samples were above the Canadian criteria, 8 were above the United States criteria, and none were above the Great Lakes sport fish consumption advisory (Table 1). We suspect that the mallards were resident individuals that had nested earlier near or in southern Green Bay. This conclusion is based on the collection date (June 12<sup>th</sup>) which is earlier than the Fall migration. Additionally, many of the birds collected were paired.

One lesser scaup was obtained during the June 12<sup>th</sup> collection (Table 2). We suspect that this individual was injured or sick and did not migrate in the fall of 1996. If that individual was a resident in Green Bay, PCB concentrations in tissues suggest that >8 months (September 1996

to June 1997) exposure to contaminants from prey items in Green Bay brought its muscle PCB concentrations above the Canadian and United States PCB poultry consumption advisories. Concentrations of PCBs in the breast muscle alone did not exceed the Great Lakes sport fish consumption advisory. However, when the breast muscle of this individual was analyzed with the associated skin, PCB concentrations did exceed the Great Lakes sport fish consumption advisory.

The results suggest limited PCB exposure to hunters consuming migrating diving ducks shot near Point au Sable, especially if breast muscle is consumed without skin attached. PCB concentrations in breast muscles of only two of 34 diving ducks collected from Point au Sable during October and November (Tables 1, 3, and 4) were above Canadian consumption guidelines, United States consumption guidelines, and the Great Lakes sport fish consumption advisory. When skin was added to the muscle (n=23), 13 samples were above the Canadian consumption guidelines, 4 above United States consumption guidelines, and none above the Great Lakes sport fish consumption advisory. The data suggest that the time period from arrival of diving ducks in Green Bay until collection (late-October to mid-November 1997) was too short to allow significant accumulation of PCBs.

Based on United States PCB consumption guidelines for poultry, mergansers shot in Lake Michigan in northern Door County should not be eaten. Of 14 diving ducks collected in Lake Michigan near the northern end of Door County in September and November, the breast muscle of 13 were above Canadian and United States consumption guidelines (Tables 1, 5, and 6). One individual was above the Great Lakes sport fish consumption advisory. Based on actively growing flight feathers, this immature female common merganser was raised locally.

Concentrations of total PCBs in muscle with skin attached are probably representative of PCB concentrations in whole carcasses. The ratio of PCB wet weight in muscle with skin to PCB wet weight in muscle without skin averaged 4.2 (range 1.5 to 7, n=8). This is very similar to the PCB breast muscle to carcass ratio (mean = 4.1, range =3.3 to 4.8) of sentinel mallards measured in another study (Custer *et al.* 1996).

### **Conclusions**

These results suggest that resident waterfowl in Green Bay accumulate PCBs to concentrations above the human consumption advisory for poultry in Canada and the United States. Tissues of migrating diving ducks shot in early fall and winter in Green Bay are generally not above human consumption advisory levels for PCBs. Based on PCB concentrations in tissues, mergansers shot in Lake Michigan near Door County should not be eaten.

### **Acknowledgments**

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## References

- Anderson, H. A., J. F. Amrhein, P. Shubat, and J. Hesse. 1993. Protocol for a uniform Great Lakes sport fish consumption advisory. Great Lakes Fish Advisory Task Force. 81 pp
- Ankley, G. T., K. B. Lodge, D. J. Call, M. D. Balcher, L. T. Brooke, P. M. Cook, R. G. Kreis, A. R. Carlson, R. D. Johnson, G. J. Niemi, R. A. Hoke, C. W. West, J. P. Giesy, P. D. Jones, and Z. C. Fuying. 1992. Integrated assessment of contaminated sediments in the lower Fox River and Green Bay, Wisconsin. *Ecotoxicol Environ Safety* 23:46-63.
- Ankley, G. T., G. J. Niemi, K. B. Lodge, H. J. Harris, D. L. Beaver, D. E. Tillitt, T. R. Schwartz, J. P. Giesy, P. D. Jones and C. Hagley. 1993. Uptake of planer polychlorinated biphenyls and 2,3,7,8-substituted polychlorinated dibenzofurans and dibenzo-p-dioxins by birds nesting in the lower Fox River and Green Bay, Wisconsin, USA. *Arch. Environ. Contam. Toxicol.* 24:332-344.
- Brieger, G., and R. D. Hunter. 1993. Uptake and depuration of PCB 77, PCB 169, and hexachlorobenzene by zebra mussels (*Dreissena polymorpha*). *Ecotoxicol. Environ. Safety* 26:153-165.
- Busch, D., and B. Schuchardt. 1991. The use of the freshwater mussel *Dreissena polymorpha* (Pallas) for biomonitoring heavy metals in limnic ecosystems: the Weser (FRG). *Verh. Int. Verein. Limnol.* 24:2261-2262.
- Carney, S. M. 1964. Preliminary keys to waterfowl age and sex identification by means of wing plumage. U. S. Fish and Wildl. Serv. Spec. Sci. Rpt. 82.
- Custer, C. M., and T. W. Custer. 1996. Food habits of diving ducks in the Great Lakes after the zebra mussel invasion. *J. Field Ornithol.* 67:86-99.
- Custer, C. M., and T. W. Custer. 1997. Occurrence of zebra mussels in near-shore areas of western Lake Erie. *J. Great Lakes Res.* 23:108-115.
- Custer T. W., and C. M. Custer. 1995. Transfer and accumulation of organochlorines from black-crowned night-heron eggs to chicks. *Environ Toxicol Chem* 14:533-536.
- Custer, T. W., D. W. Sparks, S. A. Sobiech, R. K. Hines and M. J. Melancon. 1996. Organochlorine accumulation by sentinel mallards at the Winston-Thomas sewage treatment plant, Bloomington, Indiana. *Arch. Environ. Contamin. Toxicol.* 30:163-169
- Custer C. M., T. W. Custer, P. D. Allen, K. L. Stromborg, and M. J. Melancon. 1998. Reproduction and environmental contamination in tree swallows nesting in the Fox River drainage and Green Bay, Wisconsin, USA. *Environ Toxicol Chem.* 17:1786-1798.

Custer, T. W., C. M. Custer, R. K. Hines, S. Gutreuter, K. L. Stromborg, P. D. Allen, and M. J. Melaneon. 1999. Organochlorine contaminants and reproductive success of double-crested cormorants from Green Bay, Wisconsin, USA. *Environmental Toxicology and Chemistry*. In press.

de Kock, W. C., and C. T. Bowmer. 1993. Bioaccumulation, biological effects, and food chain transfer of contaminants in the zebra mussel (*Dreissena polymorpha*). Pp. 503-533, in T. F. Nalepa and D. W. Schloesser, eds. *Zebra mussels biology, impacts, and control*. Lewis Publishers, Boca Raton, Florida.

FDA, United States Food and Drug Administration (1979) Polychlorinated biphenyls (PCBs); reduction of tolerances. *Fed Reg* 49(Jun):38330-38340

Government of Canada. 1991. *Toxic Chemicals in the Great Lakes and Associated Effects*. Vol. 1. Contaminant Levels and Trends. Environment Canada. Dept. fisheries and Oceans, Health and Welfare Canada.

Hamilton, D. J., D. Ankney, and R. C. Bailey. 1994. Predation of zebra mussels by diving ducks: an enclosure study. *Ecology* 75:521-531.

Harris, H. J., T. C. Erdman, G. T. Ankley, and K. B. Lodge. 1993. Measures of reproductive success and polychlorinated biphenyl residues in eggs and chicks of Forster's terns on Green Bay, Lake Michigan, Wisconsin-1988. *Arch Environ Contam Toxicol* 25:304-314.

Health and Welfare Canada (1991) Table 2, Division 15, Canadian Food and Drug Regulations

Hermanson M. H., E. R. Christensen, D. J. Buser, and S. M. Chen. 1991. Polychlorinated biphenyls in dated sediment cores from Green Bay and Lake Michigan. *J Great Lakes Res* 17:94-108.

Hoffman D. J., G. J. Smith, and B. A. Rattner. 1993. Biomarkers of contaminant exposure in common terns and black-crowned night herons in the Great Lakes. *Environ Toxicol Chem* 12:1719-1732.

Kovalak, W. P., G. D. Longton, and R. D. Smithee. 1993. Infestation of power plant water systems by the zebra mussel (*Dreissena polymorpha Pallas*). Pp. 359-380, in T. F. Nalepa and D. W. Schloesser, eds. *Zebra mussels biology, impacts, and control*. Lewis Publishers, Boca Raton, Florida.

Kubiak T. J., H. J. Harris, L. M. Smith, T. R. Schwartz, D. L. Stalling, J. A. Trick, L. Sileo, D. E. Docherty, and T. C. Erdman. 1989. Microcontaminants and reproductive impairment of the Forster's tern on Green Bay, Lake Michigan-1983. *Arch Environ Contam Toxicol* 18:706-727.

Leach, J. H. 1993. Impacts of the zebra mussel (*Dreissena polymorpha*) on water quality and fish spawning reefs in the western Lake Erie. Pp. 381-397, in T. F. Nalepa and D. W. Schloesser, eds. *Zebra mussels biology, impacts, and control*. Lewis Publishers, Boca Raton, Florida.

Manchester-Neesvig J. B., A. W. Andren, and D. N. Edgington. 1996. Patterns of mass sedimentation and of deposition of sediment contaminated by PCBs in Green Bay. *J Great Lakes Res* 22:444-462.

Mazak, E. J., H. J. MacIsaac, M. R. Servos, and R. Hesslein. 1997. Influence of feeding habits on organochlorine contaminant accumulation waterfowl on the Great Lakes. *Ecol. Applic.* 7:1133-1143.

Mersch, J., A. Jeaujean, H. Spor, and J. C. Pihan. 1992. The freshwater mussel *Dreissena polymorpha* as a bioindicator for trace elements, organochlorines and radionuclides. *Limnologie Aktuell* 4:227-244.

Rattner, B. A., M. J. Melancon, T. W. Custer, R. L. Hothem, K. A. King, L. J. LeCaptain, J. W. Spann, B. R. Woodin, and J. J. Stegeman. 1993. Biomonitoring environmental contamination with pipping black-crowned night heron embryos: Induction of cytochrome P450. *Environ. Toxicol. Chem.* 12:1719-1732.

Smith, V. E., J. M. Spurr, J. C. Filkins, and J. J. Jones. 1985. Organochlorine contaminants of wintering ducks foraging on Detroit River sediments. *J. Great Lakes Res.* 11:231-246.

Sullivan, J. R., J. J. Delfino, C. R. Buelow, and T. B. Sheffy. 1983. Polychlorinated biphenyls in the fish and sediment of the lower Fox River, Wisconsin. *Bull Environ Contam Toxicol* 30:58-64.

Velleux, M., and D. Endicott. 1994. Development of a mass balance model for estimating PCB export from the lower Fox River to Green Bay. *J Great Lakes Res* 20:416-434.

Wormington, A., and J. H. Leach. 1992. Concentrations of migrant diving ducks at Point Pelee National Park, Ontario, in response to invasion of zebra mussels, *Dreissena polymorpha*. *Can. Field-Nat.* 106:376-380.

Figure 1. Locations (hatched ellipses) in Green Bay and Lake Michigan where waterfowl were collected during June to November, 1997.

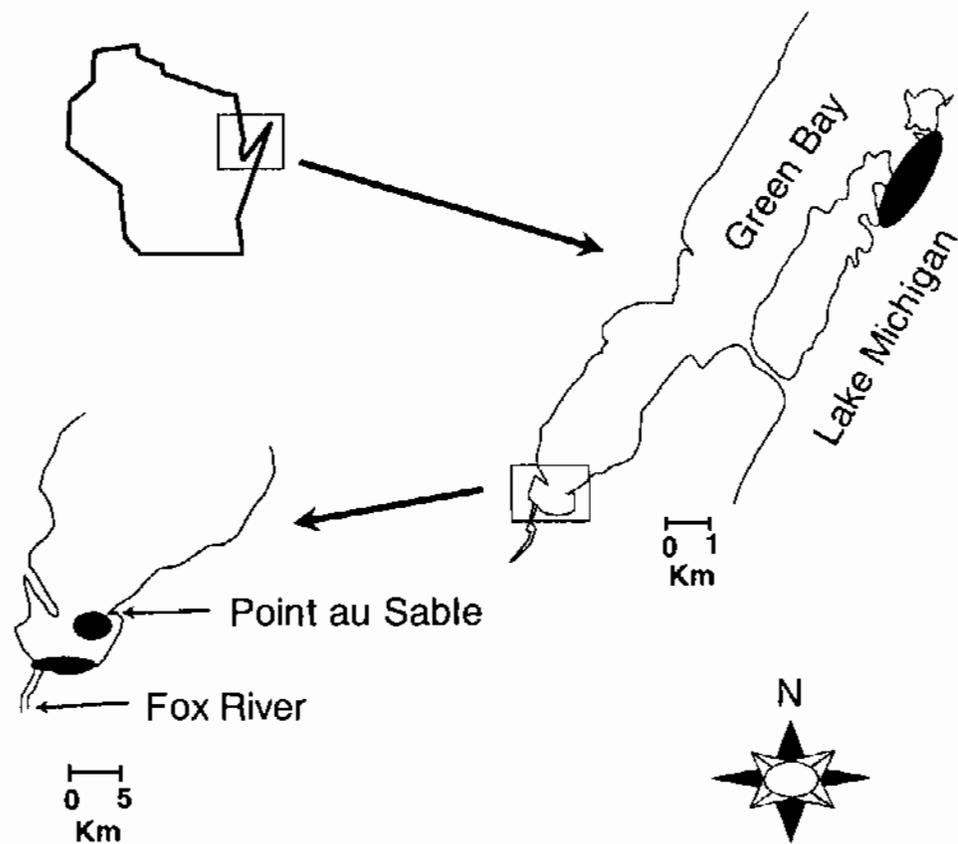


Table 1. Summary of the number of waterfowl collected near Green Bay, Wisconsin that exceeded PCB human consumption advisory levels for poultry in Canada (0.05 µg/g lipid weight), poultry in the United States (3.0 µg/g lipid weight), and fish in the Great Lakes (1.9 µg/g wet weight).

Location	No. of ducks with PCB concentrations exceeding human health criteria							
	No.	Breast muscle			No.	Breast muscle with skin attached		
		Canada	U.S.	Great Lakes		Canada	U.S.	Great Lakes
Southern Green Bay	10	5	4	0	10	10	8	0
Point au Sable	34	2	2	2	23	13	4	0
Door County	14	13	13	1	-- <sup>1</sup>	--	--	--

<sup>1</sup> -- = not measured

Table 2. PCB concentrations ( $\mu\text{g/g}$  lipid weight and  $\mu\text{g/g}$  wet weight) in skin and breast muscle of one lesser scaup and ten mallards collected in southern Green Bay on June 12, 1997. Level of detection was  $0.02 \mu\text{g/g}$  wet weight.

Cat.ID	Species	Sex	Age	PCBs $\mu\text{g/g}$ lipid weight			PCBs $\mu\text{g/g}$ wet weight		
				Muscle	Skin	Skin + muscle	Muscle	Skin	Skin + muscle
GBMD01	Mallard	M <sup>1</sup>	A <sup>2</sup>	ND <sup>3</sup>	2.8	2.0	ND	1.0	0.1
GBMD02	Mallard	F	A	ND	3.3	2.2	ND	1.2	0.1
GBMD03	Mallard	F	A	ND	11.0	8.0	ND	3.4	0.3
GBMD04	Mallard	M	A	ND	6.2	4.5	ND	2.9	0.3
GBMD05	Mallard	M	A	15.0	21.2	19.5	0.2	6.6	0.8
GBMD06	Mallard	M	A	6.6	21.5	15.4	0.1	5.1	0.6
GBMD07	Mallard	M	A	13.9	18.7	17.4	0.2	5.2	0.8
GBMD08	Mallard	F	A	2.9	11.0	9.6	0.4	4.2	0.6
GBMD09	Mallard	M	A	ND	15.5	5.6	ND	1.8	0.2
GBMD10	Mallard	M	A	5.9	22.0	16.9	0.1	6.0	0.7
GBLS11	Lesser scaup	M	A	16.3	27.9	23.3	0.6	9.5	2.0

<sup>1</sup> M = male, F = female

<sup>2</sup> A = adult

<sup>3</sup> ND = indicates not detected

Table 3. PCB concentrations ( $\mu\text{g/g}$  lipid weight and  $\mu\text{g/g}$  wet weight) in skin and breast muscle of diving ducks collected from Point au Sable, southern Green Bay on October 27, 1997. Level of detection was 0.01  $\mu\text{g/g}$  wet weight.

Cat.ID	Species	Sex	Age	PCBs $\mu\text{g/g}$ lipid weight			PCBs $\mu\text{g/g}$ wet weight		
				Muscle	Skin	Skin + muscle	Muscle	Skin	Skin + muscle
GBLS12	Greater scaup	M <sup>1</sup>	I <sup>2</sup>	ND <sup>3</sup>	2.0	1.6	ND	1.1	0.2
GBLS13	Greater scaup	F	I	ND	3.6	3.1	ND	1.7	0.3
GBLS14	Greater scaup	M	I	ND	1.2	0.8	ND	0.3	0.05
GBLS15	Greater scaup	F	I	ND	0.4	0.3	ND	0.2	0.03
GBLS16	Greater scaup	F	I	ND	2.6	2.2	ND	1.7	0.4
GBLS18	Greater scaup	M	I	ND	0.2	0.2	ND	0.2	0.04
GBLS17	Lesser scaup	F	A	ND	3.1	2.7	ND	1.9	0.4
GBLS19	Lesser scaup	M	I	ND	0.6	0.6	ND	0.5	0.2
GBCN20	Canvasback	M	I	ND	- <sup>4</sup>	-	ND	-	-
GBRD21	Ruddy duck	F	I	ND	-	-	ND	-	-
GBGE22	Common goldeneye	M	A	14.5	13.5	14.1	0.25	1.4	0.4

<sup>1</sup> M = male, F = female

<sup>2</sup> I = immature, A = adult

<sup>3</sup> ND = not detected

<sup>4</sup> - indicates no analysis

Table 4. PCB concentrations ( $\mu\text{g/g}$  lipid weight and  $\mu\text{g/g}$  wet weight) in skin and breast muscle of diving ducks collected from Point au Sable, southern Green Bay on November 12-13, 1997. Level of detection was  $0.01 \mu\text{g/g}$  wet weight.

Cat.ID	Species	Sex	Age	PCBs $\mu\text{g/g}$ lipid weight			PCBs $\mu\text{g/g}$ wet weight		
				Muscle	Skin	Skin + muscle	Muscle	Skin	Skin + muscle
GBLS23	Greater scaup	M <sup>1</sup>	A <sup>2</sup>	ND <sup>3</sup>	3.8	3.3	ND	2.2	0.4
GBLS24	Lesser scaup	M	A	ND	2.6	2.4	ND	2.0	0.6
GBLS25	Lesser scaup	M	I	ND	1.3	1.2	ND	1.1	0.4
GBLS26	Lesser scaup	M	A	4.8	5.1	5.1	0.11	2.8	0.7
GBLS27	Lesser scaup	M	I	ND	3	2.5	ND	1.8	0.3
GBLS28	Lesser scaup	F	I	ND	1.5	1.4	ND	1.3	0.6
GBLS29	Lesser scaup	M	I	ND	0.4	0.4	ND	0.3	0.1
GBLS30	Lesser scaup	M	I	ND	0.9	0.8	ND	0.7	0.2
GBLS31	Lesser scaup	M	I	ND	1.0	0.9	ND	0.7	0.2
GBBH32	Bufflehead	F	I	ND	- <sup>4</sup>	-	ND	-	-
GBBH33	Bufflehead	F	I	ND	0.5	0.4	ND	0.4	0.1
GBBH34	Bufflehead	F	I	ND	-	-	ND	-	-
GBBH35	Bufflehead	F	I	ND	-	-	ND	-	-
GBBH36	Bufflehead	M	I	ND	-	-	ND	-	-
GBBH37	Bufflehead	M	I	ND	-	-	ND	-	-
GBBH38	Bufflehead	M	A	ND	1.8	1.4	ND	1.4	0.4
GBBH39	Bufflehead	M	I	ND	-	-	ND	-	-
GBRD40	Ruddy duck	F	A	ND	-	-	ND	-	-
GBGE41	Common goldeneye	F	I	ND	0.1	0.1	ND	0.04	0.01
GBGE42	Common Goldeneye	F	I	ND	1.6	1.5	ND	1.2	0.3
GBGE43	Common goldeneye	M	I	ND	0.1	0.1	ND	0.1	0.02
GBWS44	White-winged Scoter	F	I	ND	-	-	ND	-	-
GBWS45	White-winged scoter	F	I	ND	-	-	ND	-	-

<sup>1</sup> M = male, F = female

<sup>2</sup> A = adult, I = immature

<sup>3</sup> ND = not detected

<sup>4</sup> - indicates no analysis

Table 5. PCB concentrations ( $\mu\text{g/g}$  lipid weight and  $\mu\text{g/g}$  wet weight) in skin and breast muscle of diving ducks collected from Baileys Harbor and Newport Beach, Lake Michigan on September 16-17, 1997. Level of detection was  $0.01 \mu\text{g/g}$  wet weight.

Cat.ID	Species	Sex	Age	PCBs $\mu\text{g/g}$ lipid weight			PCBs $\mu\text{g/g}$ wet weight		
				Muscle	Skin	Skin + muscle	Muscle	Skin	Skin + muscle
GBGE01	Common goldeneye	M <sup>1</sup>	A <sup>2</sup>	3.5	- <sup>3</sup>	0.2	-	-	-
GBRD01	Ruddy duck	F	A	4.6	-	-	0.1	-	-
GBRM01	Red-breasted merganser	F	A	ND <sup>4</sup>	8.5	5.2	ND	2.6	0.5
GBRM02	Red-breasted merganser	F	I	25.3	-	-	1.0	-	-

<sup>1</sup> M = male, F = female

<sup>2</sup> A = adult, I = immature

<sup>3</sup> - indicates no analysis

<sup>4</sup> ND = not detected

Table 6. PCB concentrations ( $\mu\text{g/g}$  lipid weight and  $\mu\text{g/g}$  wet weight) in skin and breast muscle of diving ducks collected in northern Door County in Lake Michigan on September 22<sup>nd</sup> and September 26<sup>th</sup>, 1997. Level of detection was 0.01  $\mu\text{g/g}$  wet weight.

Cat.ID	Species	Sex	Age	PCBs $\mu\text{g/g}$ lipid weight			PCBs $\mu\text{g/g}$ wet weight		
				Muscle	Skin	Skin + muscle	Muscle	Skin	Skin + muscle
GBPI49	Common merganser	M <sup>1</sup>	I <sup>2</sup>	8.3	- <sup>3</sup>	-	0.1	-	-
GBNP50	Common merganser	F	I	373.9	-	-	4.3	-	-
GBPI52	Common merganser	F	I	36.3	-	-	0.6	-	-
GBPI53	Common merganser	F	A	27.4	-	-	0.5	-	-
GBPI54	Common merganser	F	I	25.7	-	-	0.4	-	-
GBDI55	Common merganser	F	A	30.3	-	-	0.5	-	-
GBDI56	Common merganser	M	I	10.8	-	-	0.2	-	-
GBDI57	Common merganser	M	I	16.8	-	-	0.2	-	-
GBNP51	Red-breasted merganser	F	A	36.9	-	-	0.8	-	-
GBHI58	Red-breasted merganser	F	A	11.4	-	-	0.3	-	-

<sup>1</sup> M = male, F = female

<sup>2</sup> A = adult, I = immature

<sup>3</sup> - indicates no analysis

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