INJURY DETERMINATION REPORT

PCB CONCENTRATIONS IN HUDSON RIVER RESIDENT WATERFOWL

HUDSON RIVER NATURAL RESOURCE DAMAGE ASSESSMENT

HUDSON RIVER NATURAL RESOURCE TRUSTEES

STATE OF NEW YORK
U.S. DEPARTMENT OF COMMERCE
U.S. DEPARTMENT OF THE INTERIOR

PUBLIC RELEASE VERSION*

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*Names of individuals and affiliations have been removed for purposes of litigation

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INJURY DETERMINATION REPORT
PCB CONCENTRATIONS IN HUDSON RIVER RESIDENT WATERFOWL
Past and continuing discharges of polychlorinated biphenyls (PCBs) have contaminated the natural resources of the Hudson River. The Hudson River Natural Resource Trustees (HRNRT) – New York State, the U.S. Department of Commerce, and the U.S. Department of the Interior – are conducting a natural resource damage assessment (NRDA) to assess and restore those natural resources injured by PCBs (HRNRT 2002). Waterfowl represent an important ecological and recreational resource on the Hudson River. As part of the assessment process, the Trustees investigated PCB contaminant levels in Hudson River resident waterfowl in order to determine whether certain Hudson River waterfowl have been injured by PCBs found in the environment. The term “resident” as used in this report refers to mallards ducks (*Anas platyrhynchos*) that have bred, nested, or hatched on the Hudson River in a given year.

Potential injuries to resident waterfowl from PCBs include the presence of PCB concentrations in edible portions of the waterfowl that exceed tolerance levels enforced by the U.S. Food and Drug Administration (USFDA) (43 CFR 11.62(f)(1)(ii)). The federal tolerance for PCBs in poultry is 3.0 parts per million (ppm or μg/g wet weight) in fat (21 CFR 109.30(a)(3)). The USFDA defines poultry as any domesticated bird, including ducks (9 CFR 381.1), and any migratory waterfowl or game bird (9 CFR 362.1(d)).

Among migratory ducks, mallards are the most numerous species of duck on the Hudson River. After breeding in early spring, adult mallards have a period during the summer when they are flightless and take up residency on the river for several months. When hatched, juvenile mallards are flightless and remain flightless and resident to the river for most of the summer, until the fall migration. During this time of residency, essentially all chemical exposures are local, and juvenile birds have spent their entire lives as residents of the river. This study collected mallards during a time when ducks were non-migratory and often flightless, ensuring that ducks were resident to the Hudson River.

The Hudson River PCB Superfund Site remedy was selected in 2002, and the first phase of remediation commenced in 2009. Resident waterfowl sampling was conducted prior to remedy implementation. From July 21, 2008 to August 28, 2008, mallards were collected from four study areas on the Hudson River:

- **Area 1** – A reference area upstream of South Glens Falls in the upper river between the Feeder Canal Dam and International Paper Company in Corinth (River Mile (RM) 220 to RM 204), and Great Sacandaga Lake;
- **Area 2** – Fort Edward to Northumberland in the upper river (including the Thompson Island Dam; RM 194 to RM 182);
- **Area 3** – Schuylerville to Mechanicville in the upper river (RM 180 to RM 160); and
- **Area 4** – New Baltimore to Newburgh in the lower river (RM 132 to RM 56).
A subset of mallards from each study area was analyzed for PCBs. Muscle and fat samples were analyzed for approximately 10 adult male, 10 adult female, and 20 juvenile mallards for study areas 1, 2 and, 3. Muscle and fat samples were analyzed for 5 adult male, 5 adult female, and 10 juvenile mallards for Area 4.

Areas 2 and 3 downstream of the General Electric (GE) plant sites and above the Federal Dam at Troy, had significantly higher PCB concentrations in both muscle and fat than the reference area. Area 4, the Hudson River estuary, had significantly lower PCB concentrations than Areas 2 and 3, but significantly higher PCB concentrations than the reference area. PCB concentrations in mallards collected between Ft. Edward and Mechanicville were approximately 100 times greater than PCB concentrations in mallards collected from the reference area. The data demonstrate that juvenile and adult mallards rapidly accumulated PCBs from the PCB-contaminated stretches of the Hudson River during their residency on the river.

One hundred percent (100%) of the mallard fat samples from Area 2, ninety-two (92%) from Area 3, and 5% from Area 4 exceeded the federal tolerance of 3 ppm compared to 3% of samples upstream of the GE plant sites. The percentage of fat samples exceeding the tolerance value in Areas 2 and 3 was significantly higher than in Area 1.

PCB concentrations in sediments, water, and biota have generally declined since the enactment of CERCLA. It is reasonable to assume equal or higher concentrations in Hudson River waterfowl during the years 1981-2007 based on the results of the 2008 waterfowl study. The data demonstrate that resident mallards accumulated PCBs from the Hudson River at levels that exceed the federal tolerance for PCB levels in poultry. Therefore, an injury to a biological resource, waterfowl resident to the Hudson River, as defined in 43 CFR 11.62(f)(1)(ii), has occurred.
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<th>Description</th>
</tr>
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</tr>
<tr>
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</tr>
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</table>
# Acronyms and Abbreviations

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>CERCLA</td>
<td>Comprehensive Environmental Response, Compensation, and Liability Act</td>
</tr>
<tr>
<td>EPA</td>
<td>U.S. Environmental Protection Agency</td>
</tr>
<tr>
<td>GE</td>
<td>General Electric Company</td>
</tr>
<tr>
<td>HRNRT</td>
<td>Hudson River Natural Resource Trustees</td>
</tr>
<tr>
<td>NRDA</td>
<td>Natural Resource Damage Assessment</td>
</tr>
<tr>
<td>NYSDOH</td>
<td>New York State Department of Health</td>
</tr>
<tr>
<td>PCBs</td>
<td>Polychlorinated biphenyls</td>
</tr>
<tr>
<td>ppb</td>
<td>Parts per billion (for example, one microgram per kilogram, or (\mu g/\text{kg}))</td>
</tr>
<tr>
<td>ppm</td>
<td>Parts per million (for example, one milligram per kilogram, or mg/kg)</td>
</tr>
<tr>
<td>tPCBs</td>
<td>Total polychlorinated biphenyls</td>
</tr>
<tr>
<td>USFDA</td>
<td>U.S. Food and Drug Administration</td>
</tr>
<tr>
<td>ww</td>
<td>Wet weight</td>
</tr>
</tbody>
</table>
1.0 INTRODUCTION

1.1 BACKGROUND

Past and continuing discharges of polychlorinated biphenyls (PCBs) have contaminated the natural resources of the Hudson River. The Hudson River Natural Resource Trustees (HRNRT) – New York State, the U.S. Department of Commerce, and the U.S. Department of the Interior – are conducting a natural resource damage assessment (NRDA) to assess and restore those natural resources injured by PCBs (HRNRT 2002). Waterfowl represent an important ecological and recreational resource on the Hudson River. As part of the assessment process, the Trustees investigated PCB contaminant levels in Hudson River resident waterfowl in order to determine whether certain Hudson River waterfowl have been injured by PCBs found in the environment. The term “resident” as used in this report refers to mallards that have bred, nested, or hatched on the Hudson River in a given year. The term “juvenile” as used in this report refers to resident mallards that were hatched on the Hudson River in a given year.

1.2 POLYCHLORINATED BIPHENYLS (PCBS)

Polychlorinated biphenyls, also known as PCBs, have polluted large stretches of the Hudson River since the 1940s. EPA has estimated that the two General Electric (GE) manufacturing facilities located in Fort Edward and Hudson Falls, New York, discharged up to 1.3 million pounds of PCBs into the river between the 1940s and 1977 (USEPA 1991). PCBs persist in the environment for many decades, and scientific research indicates they can be harmful to animals and humans. Adverse effects, including partial or complete reproductive failure, birth defects, impaired growth, behavioral changes, lesions, immune system dysfunction, and hormone imbalances, have been observed in a wide variety of species, including fish, amphibians, birds, and mammals (e.g., Barron et al. 2000, Harris et al. 1995, Monosson 1999, Rosenshield et al. 1999, Sparling 2010, Tillitt et al. 1993, Zwiernik et al. 2011). The primary route of human exposure to PCBs is through the consumption of contaminated fish and wildlife species (Carpenter 2006).

1.3 RESIDENT WATERFOWL

Among migratory waterfowl, mallards (Anas platyrhynchos) are the most abundant duck species in North America (Drilling et al. 2002) and constitute greater than 50% of the total breeding population of all ducks in the northeast (USFWS 2012). Mallards are also the most numerous duck species in New York (Stegemann and Swift 2008). The Hudson River is part of the Hudson-Champlain corridor, one of the major migratory waterfowl pathways in New York State. In the Hudson River basin, mallards typically arrive from spring migration in March or April, and nesting and brooding of eggs occurs by mid- to late April. The young usually hatch in mid-May to early June. At hatch, waterfowl are flightless, and juveniles remain flightless until about mid-July when flight feathers develop. In early summer, following the breeding period, the adults undergo molting of their flight feathers and are flightless for a period of about two months (Drilling et al. 2002).

Waterfowl can rapidly accumulate PCBs in their tissues (Foley and Batcheller 1988, Skinner 1992, Swift et al. 1993). Between the spring breeding season and departure with the fall migration, waterfowl on the Hudson River spend approximately five months as residents on the river, during which time they are exposed to PCB contamination. Several previous studies have measured PCB concentrations in various waterfowl species taken from the Hudson River and found concentrations ranging from <0.1 ppm to as high as 43 ppm depending on the species and the tissue sampled (Baker et al. 1976, Foley 1992, Kim et al. 1985, Kim et al. 1984, O’Keefe et al. 2006). In most of these studies, sample sizes were small, and collections occurred within the hunting season during fall migrations. Once fall migration begins, waterfowl from other areas co-mingle
with resident Hudson River birds. At this time Hudson River and migratory waterfowl of the same species cannot be readily distinguished from each other. This study collected mallards during a time when ducks were non-migratory and often flightless, ensuring that ducks were resident to the Hudson River.

The Hudson River from Hudson Falls to the Federal Dam at Troy is segmented into “pools” by a series of dams. Based on observations from New York State Department of Environmental Conservation biologists, the ducks within each pool tend to remain within that pool on the river from the time of nesting and brooding through the summer and before autumn migration. As a consequence, essentially all chemical exposures to juvenile birds are local. The only exception may be the residues that have been transferred to the young by the adult female via maternal deposition into the egg. During the summer flightless period, adults also rely solely on food sources within the river and maintain a close association with their young to support their continued growth.

1.4 DEFINITION OF INJURY

Pursuant to the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the U.S. Department of the Interior has promulgated regulations that define a number of categories of injuries to natural resources (43 CFR 11.10 et seq.). Potential injuries to resident waterfowl from PCBs include the presence of PCB concentrations in edible portions of the waterfowl that exceed tolerance levels promulgated by the U. S. Food and Drug Administration (USFDA) (43 CFR 11.62(f)(1)(ii)). The USFDA defines poultry as any domesticated bird, including ducks (9 CFR 381.1), and any migratory waterfowl or game bird (9 CFR 362.1(d)). The federal tolerance level for PCBs in poultry is 3.0 parts per million (ppm or μg/g) in fat (21 CFR 109.30(a)(3)).

2.0 METHODS

2.1 MALLARD COLLECTION

Juvenile and adult mallards were collected between July 21, 2008 and August, 28, 2008 in the year prior to remedy implementation from four areas of the Hudson River (Figure 1):

Area 1 - A reference area upstream of South Glens Falls in the upper river between the Feeder Canal Dam and International Paper Company in Corinth (River Mile (RM) 220 to RM 204); and Great Sacandaga Lake;

Area 2 - Fort Edward to Northumberland in the upper river (including the Thompson Island Pool; RM 194 to RM 182);

Area 3 - Schuylerville to Mechanicville in the upper river (RM 180 to RM 160); and

Area 4 - New Baltimore to Newburgh in the lower river (RM 132 to RM 56).

Mallards were collected by shotgun from each of the four areas. The target numbers for mallards were 15 adult females, 15 adult males, and 30 juveniles, but actual numbers varied among study areas (Table 1). Adult and juvenile collections occurred during July and August to coincide with the flightless stage and ensure that ducks were resident to the Hudson River. Mallard eggs were collected opportunistically between April 14, 2008 and May 30, 2008.

2 The Hudson River flows from River Mile 315 at Lake Tear of the Clouds, in the Adirondack Mountains, south to the Battery in Manhattan. “River Miles” are miles upstream from the Battery, which is designated “River Mile 0.”
TABLE 1. NUMBER OF MALLARDS COLLECTED FROM FOUR STUDY AREAS ALONG THE HUDSON RIVER, NY IN JULY AND AUGUST, 2008

<table>
<thead>
<tr>
<th>Area</th>
<th>Adult Female</th>
<th>Male</th>
<th>Juvenile</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area 1</td>
<td>22</td>
<td>15</td>
<td>27</td>
</tr>
<tr>
<td>Area 2</td>
<td>20</td>
<td>15</td>
<td>23</td>
</tr>
<tr>
<td>Area 3</td>
<td>15</td>
<td>13</td>
<td>31</td>
</tr>
<tr>
<td>Area 4</td>
<td>10</td>
<td>16</td>
<td>26</td>
</tr>
</tbody>
</table>

Area 1 = a reference area between the Feeder Canal Dam and International Paper Company in Corinth, and Great Sacandaga Lake; Area 2 = Fort Edward to Northumberland; Area 3 = Schuylerville to Mechanicville; and Area 4 = New Baltimore to Newburgh.

After collection, specimens were labeled, weighed, and sexed in the field before being transported under chain of custody procedure to the NYS Department of Environmental Conservation (DEC) Hale Creek Field Station. During processing adult and juvenile mallard breast muscle and subcutaneous fat samples were removed and placed into chemically clean jars. For egg samples, egg contents were removed and placed into chemically clean jars. All samples were stored under chain of custody procedure in a locked freezer at -20°C until they were shipped to the analytical laboratory. A more detailed description of the collection and sample processing methods followed is available in the Study Plan for Waterfowl Injury Assessment: Determining PCB Concentrations in Hudson River Resident Waterfowl (HRNRT 2008), and the standard operating procedures for collection and processing waterfowl samples are in Appendix A. Appendix B lists collection information on the mallards analyzed.

2.2 SAMPLE SELECTION

Specimens were selected to provide equal numbers in each category across study areas to the extent possible (e.g., male and female; adult and juvenile) with preference given to specimens that had adequate sample mass for both fat and muscle. Forty mallards from Areas 1, 2, and 3 were targeted for analysis: 10 adult females, 10 adult males, 20 juveniles. Twenty samples from Area 4 were chosen for analysis: 5 adult females, 5 adult males, and 10 juveniles (Figure 2). Not all categories met target sample sizes (Table 2). Typically, a muscle and fat sample from each mallard specimen was analyzed. However, six of the juvenile mallards from Area 2 did not have enough dissectible subcutaneous fat for an adequate sample mass. One of the two additional juvenile mallards with enough mass for both muscle and fat samples was added to the analysis. Although the sex of juvenile birds was noted upon collection, juvenile birds from each study area were pooled for statistical analyses regardless of sex. Prior to reaching sexual maturity, physiological differences between males and females that might influence differing rates of PCB accumulation (e.g., fat storage, egg development, etc.) are expected to be minimal. Appendix B lists collection information on the mallards selected for chemical analysis. Although the four eggs collected were analyzed, the small sample size and limited spatial distribution prevented robust statistical analysis. Therefore results from egg samples are not included in this report. Appendix E, however, includes results from the chemical analysis of these eggs.
Table 2. Number of Mallard Muscle and Fat Samples Analyzed from Mallards Collected on the Hudson River, NY in July and August, 2008

<table>
<thead>
<tr>
<th></th>
<th>Area 1</th>
<th>Area 2</th>
<th>Area 3</th>
<th>Area 4</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Muscle</td>
<td>Fat</td>
<td>Muscle</td>
<td>Fat</td>
</tr>
<tr>
<td>Adult</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Male</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Female</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Juvenile</td>
<td>18</td>
<td>18</td>
<td>21</td>
<td>15</td>
</tr>
</tbody>
</table>

2.3 Chemical Analyses

Chemical analyses for breast muscle and subcutaneous fat samples included total PCBs, PCB homologs, the 209 PCB congeners, moisture, and lipid content. Total PCB, PCB homologs, and PCB congeners were analyzed using low resolution mass spectrometry, and percent lipid and moisture were measured via gravimetric methods, as specified in the Analytical Quality Assurance Plan (HRNRT 2005).

2.4 Quality Assurance/Quality Control

Analytical chemistry data were validated as specified in the Analytical Quality Assurance Plan (HRNRT 2005) and the Study Plan for Waterfowl Injury Assessment: Determining PCB Concentrations in Hudson River Resident Waterfowl (HRNRT 2008). Sample results and related QC data were received in both an electronic and hard copy format. Electronic data were verified against the hard copy data package. All data packages received a summary validation (Level III).

2.5 Statistical Analysis

Statistics were used to answer these questions:

1) Do resident waterfowl in study areas downstream of the GE plants have higher PCB concentrations than waterfowl from areas upstream of the plants?

2) Do PCB concentrations in waterfowl fat exceed the USFDA tolerance of 3.0 parts per million (fat basis)?

3) What proportions of mallard samples exceed the federal tolerance?

To answer these questions, the data were summarized using descriptive statistics and tabulated to determine the proportion of samples that exceed the federal tolerance. The distribution of PCB homolog groups expressed as a fraction of tPCBs was used to compare differences in homolog patterns between study areas. A Kruskal-Wallis (KW) test with Monte Carlo methods was used to compare PCB concentrations in mallards collected from each area of the river. Where KW analysis detected statistically significant differences among parameters, pairwise comparisons were made using Kolmogorov-Smirnov (KS) two-sample tests with Monte Carlo methods and a Bonferroni correction. Fisher’s Exact test (FE) was used to determine spatial differences in proportional exceedance of the FDA tolerance for PCB in poultry. Probabilities less than 0.05 were considered significant. All statistical analyses were calculated using Cytel Studio STATXACT 8.
3.0 RESULTS

The 2008 Waterfowl Data Sheets provide the analytical results (Appendix E). The 2008 Waterfowl Data Sheets contains the following fields:

Sampling Date - mm/dd/yy

Field ID - Field IDs were created using the format: A2MAL-JM-01
Where “A2” = the study area; “MAL” = mallard; “J” = juvenile (A=adult, E=egg); “M”=male (F=female); and “01”=specimen number.

Matrix – “Matrix” refers to the tissue sample type: muscle, fat, or egg contents.

Laboratory ID - "Laboratory ID" refers to the ID assigned by Axys Analytical Services Ltd. (Axys) to samples analyzed for PCBs.

Easting - NAD83 Universal Transverse Mercator easting coordinates (meters) Zone 18N.

Northing - NAD83 Universal Transverse Mercator northing coordinates (meters) Zone 18N.

Analyte - For the PCB congeners, the analyte names are reported using the following format: PCB-NNN
NNN refers to the International Union of Pure and Applied Chemistry (IUPAC) congener number. For example, PCB 126 is reported as PCB-126. For co-eluting PCB congeners, a combined analyte name was used (e.g., PCB-5/8 indicates the combined value detected for PCB-5 and PCB-8).

The total concentration of all congeners within a chlorination level is represented by the chlorination level name. For example, the total concentration of all pentachlorinated biphenyls is reported as: Total Pentachlorobiphenyls. When a congener is not detected, its contribution to the total is counted as zero. For all other analytes, the analyte name is reported in this field. Total Polychlorobiphenyls is the sum of all detected congeners.

Value - Analytical result.

Interpretive Qualifier - The qualifier in this field should be used when interpreting the reported results. This field contains a combination/merge of the Lab Flag field and the DV (data validation) Qualifier field (Appendix E).

The qualifiers are defined as follows:
- U - Analyte was not detected. The associated value represents the detection limit.
- J - Estimated: The associated numerical value is an estimated quantity. The analyte was detected, but the reported value may not be accurate or precise. The “J” qualification indicates the data fell outside the QC limits, but the exceedance was not sufficient to cause rejection of the data, or the reported result is with a range of elevated analytical uncertainty (greater than the Method Detection Limit (MDL) value, but less than the Practical Quantitation Limit (PQL) value).
- UJ - Estimated/Not detected: An analysis was performed for the compound or analyte, but it was not detected and the sample quantitation or detection limit may be inaccurate or imprecise. The associated numerical result is the detection limit.

Detection Limit - The analytical limit below which an analyte cannot be detected.

Units - The unit of measurement of the analytical result (e.g., ng/g or “PERCENT”).
For the purpose of reporting PCB results in the text, tables, and figures included in this report, all values flagged with either a U or UJ qualifier (that is, not detected; see Appendix E) were considered to be zero. Using zero, rather than the value reported by the laboratory for the analyte, which represents the detection limit for the analysis, potentially underreports the true value, but avoids over-reporting the true value. The actual PCB concentrations could be higher. When results were flagged with a J qualifier, the estimated value was used.

Results in the 2008 Waterfowl Data Sheets are presented in ng/g wet weight, which is equivalent to part per billion (ppb). In the text of this report, PCB concentrations are presented in parts per million (ppm), which is equivalent to μg/g.

### 3.1 DATA VALIDATION

Of the 46,704 sample results, a total of 1,140 results were estimated because of laboratory accuracy and precision outliers. A total of 2,102 PCB congener results were qualified as not detected (U) at the reported values to indicate results that did not meet the quantitation criteria and were not positively identified. The overall quality of the data is acceptable and the results, as qualified, are considered usable. There are no rejected data in this data set; therefore, the completeness for this data set is 100%. The full Data Quality Evaluation Reports are available in Appendix C.

### 3.2 TOTAL PCB CONCENTRATIONS

**Muscle** - Total PCB (tPCB) concentrations in mallard muscle tissue collected from the Hudson River ranged from 0.000037 ppm to 1.880 ppm wet weight (Table 3). Total PCB concentrations in muscle were significantly different across study areas (KW: K=98.08, df=3, p<0.001; Figure 3). Concentrations were lowest in Study Area 1 and showed a significant increase in Areas 2 and 3 (KS: p<0.01 and p<0.01, respectively). Total PCB concentrations in muscle were significantly lower in Area 4 than in either of Area 2 and Area 3 (KS: p<0.01 and p<0.01, respectively), but were still significantly higher than in Area 1 (KS: p<0.01)

**Table 3. Total PCB Concentrations (Wet Weight) in Muscle Tissue for Mallards Collected from Four Study Areas Along the Hudson River, NY in July and August, 2008 (Juveniles and Adults Combined)**

<table>
<thead>
<tr>
<th>Study Area</th>
<th>Mean (ppm)</th>
<th>S.D.(±)</th>
<th>Min (ppm)</th>
<th>Max (ppm)</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0039</td>
<td>0.0078</td>
<td>0.0003</td>
<td>0.046</td>
<td>38</td>
</tr>
<tr>
<td>2</td>
<td>0.508</td>
<td>0.405</td>
<td>0.0937</td>
<td>1.88</td>
<td>41</td>
</tr>
<tr>
<td>3</td>
<td>0.412</td>
<td>0.322</td>
<td>0.0007</td>
<td>1.24</td>
<td>38</td>
</tr>
<tr>
<td>4</td>
<td>0.027</td>
<td>0.0314</td>
<td>0.00004</td>
<td>0.124</td>
<td>20</td>
</tr>
</tbody>
</table>

When categorized based upon age (Table 4), tPCB concentrations in juvenile and adult mallard muscle were also significantly different across study areas (KW: K=45.29, df=3, p<0.001; K=53.94, df=3, p<0.001, respectively), and exhibited similar patterns (Figure 4). Areas 2, 3, and 4 were significantly higher than Area 1.
With Areas 2 and 3 significantly higher than Area 4 (KS: p<0.01). Area 3 adults tended to have lower PCB concentration in muscle than Area 2 adults, but pairwise comparison with the Bonferroni correction was not significant.

Table 4. Total PCB concentrations in muscle tissue from juvenile (J) and adult (A) mallards collected on the Hudson River, NY in July and August, 2008

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0013</td>
<td>0.0062</td>
<td>0.0009</td>
<td>0.0103</td>
<td>18</td>
<td>0.0042</td>
<td>0.0456</td>
<td>0.00028</td>
<td>0.00059</td>
<td>20</td>
</tr>
<tr>
<td>2</td>
<td>0.472</td>
<td>0.545</td>
<td>0.393</td>
<td>0.425</td>
<td>21</td>
<td>1.740</td>
<td>1.880</td>
<td>0.094</td>
<td>0.099</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>0.523</td>
<td>0.312</td>
<td>0.362</td>
<td>0.251</td>
<td>18</td>
<td>1.240</td>
<td>0.814</td>
<td>0.00067</td>
<td>0.057</td>
<td>20</td>
</tr>
<tr>
<td>4</td>
<td>0.020</td>
<td>0.035</td>
<td>0.038</td>
<td>0.024</td>
<td>10</td>
<td>0.124</td>
<td>0.081</td>
<td>0.00004</td>
<td>0.00054</td>
<td>10</td>
</tr>
</tbody>
</table>

Fat - Total PCB concentrations in mallard fat collected from the Hudson River ranged from 0.007 ppm to 132 ppm wet weight (Table 5). Total PCB concentrations in fat were significantly different across study areas (KW: K=94.41, df=3, p<0.001). Similar to the patterns observed in muscle, PCB concentrations in mallard fat tissue were lowest in Area 1, increased significantly in Areas 2 and 3 (KS: p<0.01), before decreasing again in Area 4 (KS: p<0.01; Figure 5). Unlike the patterns in muscle, tPCB concentrations in fat from Area 3 were significantly lower than Area 2 (KS: p<0.05). Total PCB concentrations in fat from Area 4 was significantly lower than Areas 2 and 3, but significantly higher than Area 1 (KS: p<0.01).
When categorized based upon age (Table 6), tPCB concentrations in juvenile and adult mallards fat were also significantly different across study areas (KW: K=41.01, df=3, p<0.001; K=52.9, df=3, p<0.001, respectively), and exhibited similar patterns. Similar to patterns of tPCBs in muscle, both adults and juveniles from Areas 2 and 3 exhibited significantly higher tPCBs in fat than the other two study areas (KS: p<0.01, Figure 6). Mean juvenile PCB concentrations in Areas 2 and 3 were more than 300 and 200 times greater, respectively, than the mean juvenile PCB concentrations in Area 1, and more than 50 and 30 times greater, respectively, than the mean juvenile PCB concentrations in Area 4. No statistically significant difference between Area 2 and 3 tPCBs in fat were evident for juveniles. Area 3 adults tended to have lower PCB concentration in fat than Area 2, but pairwise comparison with the Bonferroni correction was not significant. Area 4 adults had significantly higher tPCB concentrations in fat than Area 1 adults (KS: p<0.05). No significant difference in tPCB concentrations in fat was evident between juveniles in Areas 1 and 4.

### Table 5. Total PCB Concentrations (Wet Weight) in Fat Tissue for Mallards Collected from Four Study Areas along the Hudson River, NY in July and August, 2008 (Juveniles and Adults Combined)

<table>
<thead>
<tr>
<th>Study Area</th>
<th>Mean (ppm)</th>
<th>S.D.(±)</th>
<th>Min (ppm)</th>
<th>Max (ppm)</th>
<th>n</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.347</td>
<td>0.992</td>
<td>0.03</td>
<td>6.174</td>
<td>38</td>
</tr>
<tr>
<td>2</td>
<td>39.15</td>
<td>32.39</td>
<td>3.480</td>
<td>132.0</td>
<td>35</td>
</tr>
<tr>
<td>3</td>
<td>24.73</td>
<td>25.53</td>
<td>0.034</td>
<td>94.57</td>
<td>38</td>
</tr>
<tr>
<td>4</td>
<td>1.306</td>
<td>1.197</td>
<td>0.007</td>
<td>4.463</td>
<td>20</td>
</tr>
</tbody>
</table>

### Table 6. Total PCB Concentrations (Wet Weight) in Fat Tissue from Juvenile (J) and Adult (A) Mallards Collected on the Hudson River, NY in July and August, 2008

<table>
<thead>
<tr>
<th>Study Area</th>
<th>Mean (ppm)</th>
<th>S.D.(±)</th>
<th>Min (ppm)</th>
<th>Max (ppm)</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>J</td>
<td>A</td>
<td>J</td>
<td>A</td>
<td>J</td>
<td>A</td>
</tr>
<tr>
<td>1</td>
<td>0.149</td>
<td>0.526</td>
<td>0.0795</td>
<td>1.356</td>
<td>18 20</td>
</tr>
<tr>
<td>2</td>
<td>50.10</td>
<td>30.95</td>
<td>33.76</td>
<td>29.54</td>
<td>15 20</td>
</tr>
<tr>
<td>3</td>
<td>34.12</td>
<td>16.28</td>
<td>32.78</td>
<td>11.27</td>
<td>18 20</td>
</tr>
<tr>
<td>4</td>
<td>0.954</td>
<td>1.657</td>
<td>1.064</td>
<td>1.273</td>
<td>10 10</td>
</tr>
</tbody>
</table>
3.3 PCB HOMOLOGS

The 209 PCB congeners were grouped into homolog groups based on chemical structure related to the number of chlorine atoms (1-10) attached to the biphenyl core. The distribution of homologs expressed as a fraction of tPCBs was different among study areas for both muscle and fat tissue (Figure 7). The homolog patterns observed in Areas 2 and 3 were dominated by tetra-, penta-, and hexa-chlorinated biphenyls, whereas the homolog patterns from Areas 1 and 4 were dominated by higher chlorinated homologs (hexa- and hepta-chlorinated biphenyls).

3.4 FEDERAL TOLERANCE VALUE

The federal tolerance value for PCBs in poultry is 3 ppm (wet weight) in fat. In Area 2, 100% of the tPCB concentrations in fat samples exceeded the tolerance value, some by as much as 40 times the tolerance value, and 92% of the fat samples from Area 3 exceeded the tolerance (Figure 8). One fat sample from Area 1 (3%) and one fat sample from Area 4 (5%) exceeded the federal tolerance value (Table 7). The percentage of mallard fat samples exceeding the tolerance value in Areas 2 and 3 was significantly different than the reference area (FE: p<0.001, p<0.001, respectively). No significant difference was detected between the percentage of fat samples exceeding the tolerance value in Area 4 and the reference area.

<table>
<thead>
<tr>
<th>Study Area</th>
<th>&lt; 3 ppm</th>
<th>&gt; 3 ppm</th>
<th>% Exceeding Tolerance</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>37</td>
<td>1</td>
<td>3</td>
<td>38</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>35</td>
<td>100</td>
<td>35</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>35</td>
<td>92</td>
<td>38</td>
</tr>
<tr>
<td>4</td>
<td>19</td>
<td>1</td>
<td>5</td>
<td>20</td>
</tr>
</tbody>
</table>

4.0 DISCUSSION

4.1 PCB CONCENTRATIONS IN HUDSON RIVER RESIDENT WATERFOWL

Several previous studies have collected waterfowl from the vicinity of the Hudson River for PCB analysis, but very little data exist for mallards. Foley et al. (1992) analyzed mallards collected from the Hudson-Champlain Corridor and found wet weight PCB values in subcutaneous fat ranging as high 22.7 ppm, which falls within the range of values found in this study. However, these data on mallards (and most data available for waterfowl) come from birds collected during the hunting season, which coincides with fall migration. During migration, there is no clear way to determine whether mallards collected on the Hudson River are resident, or whether mallards are picking up PCBs from sources other than the Hudson River.

Kim et al. (1984) and Kim et al. (1985) analyzed some fall-collected Hudson River mallards (n=16 and n=10, respectively), but all samples were from the estuary portion of the river (Area 4) with no samples in the more heavily PCB-contaminated Upper Hudson River. Limited historic data exist for mallards from the Thompson Island Pool (Area 2) collected during the flightless period. Two mallards collected in the Thompson Island Pool during August 1995 were composited as whole body samples (without beak and feet) and had a PCB concentration of 4.022 ppm (USGS 1996). During the fall collections, most sub-cutaneous
fat in a variety of waterfowl sampled exceeded the FDA tolerance of 3 ppm, but the Hudson River resident status (bred, nested or hatched) of those birds could not be confirmed (Kim et al. 1984, Kim et al. 1985). Appendix D presents a detailed table of available waterfowl data for the Hudson River.

This study collected mallards during a time when ducks were non-migratory and flightless, ensuring that ducks were resident to the Hudson River. Juvenile mallards collected during the study period were flightless and would have hatched on or adjacent to the Hudson River. Aside from any potential PCB contributions from maternal deposition, juvenile mallards collected during this study would be exposed only to local contaminant sources. Adult mallards collected during the study period would have arrived in early spring and spent several months feeding on and around the Hudson River in preparation for the breeding season. Both adult and juvenile mallards in river sections downstream of the GE plants (Areas 2 and 3) have greatly elevated levels of PCBs when compared to mallards in areas of the river upstream of the GE plants. Although mallards sampled from the Hudson River estuary (50-100 miles downstream of the GE plants) had significantly lower PCB levels than mallards in Areas 2 and 3, mallards in the estuary were still significantly more contaminated with PCBs than the reference area. Given the significant differences between resident mallard PCB concentrations in the Hudson River downstream of the GE plants, and those from the reference area and the Hudson River estuary, mallards are rapidly accumulating PCBs during the breeding season on the Upper Hudson River. The results of this study are consistent with other studies that have documented the rapid accumulation of organochlorine compounds in waterfowl from other waterbodies (Foley and Batcheller 1988, Skinner 1992, Swift et al. 1993).

The consistent homolog patterns between Areas 2 and 3 suggest a common, relatively less weathered source of PCBs, as opposed to samples from Area 1 (reference) and from the Hudson River estuary (Area 4) where the homolog pattern is shifted to slightly higher chlorinated homologs. Similar homolog patterns have been reported for Hudson River sediment, fish, and invertebrates (Sloan et al. 2005, HRNRT 2009), and suggest the direct influence of PCBs from the GE plants on PCB concentrations in Hudson River waterfowl.

4.2 SAMPLES EXCEEDING THE FEDERAL TOLERANCE

The USFDA tolerance value for PCBs in poultry is 3 ppm in fat (wet weight), and fat samples from ducks collected in study areas downstream of the GE plants exhibited large proportions of samples that exceed the federal tolerance. All of the fat samples from Area 2, which is the first river section downstream of the GE plants, exceeded the tolerance value, and ninety-two percent (92%) of the fat samples from Area 3 exceeded the tolerance value. The highest concentrations in Areas 2 and 3 exceeded the tolerance value by approximately 30 and 40 times, respectively. In contrast, one fat sample (3%) from the reference area above the GE plants and one fat sample (5%) from the Hudson River estuary exceeded the tolerance value, but both samples came from adult mallards. Kim et al. (1984) and Kim et al. (1985) also compared PCBs in fat samples from mallards collected on the Hudson River Estuary (Columbia County; Area 4) during 1979 and 1981 to the USFDA tolerance value and found that 56% of the samples collected in 1979 and 50% of the samples collected in 1981 exceeded the tolerance. Comparing the results from Kim et al. (1984 and 1985) to our study results suggests that the proportion of mallards exceeding the tolerance has decreased in the Hudson River Estuary. However, in the Upper Hudson 92-100% of the mallards sampled still exceed the USFDA tolerance value, more than 35 years after GE ceased direct discharges of PCBs to the river.
5.0 CONCLUSIONS

This study provides a substantial data set for PCB concentrations in edible tissues of resident waterfowl in the Hudson River. The data demonstrate that resident mallards are accumulating significantly higher PCB concentrations in areas downstream of the GE plants on the Hudson River when compared to a reference area. PCB concentrations in mallards collected between Ft. Edward and Mechanicville were approximately 100 times greater than PCB concentrations in mallards collected from the reference area. The data also demonstrate that resident mallards accumulated PCBs from the Hudson River at levels that exceed the federal tolerance for PCB levels in poultry. In the Upper Hudson 92-100% of the mallards sampled still exceed the USFDA tolerance value, more than 35 years after GE ceased direct discharges of PCBs to the river.

PCB concentrations in sediments, water, and biota have generally declined since the enactment of CERCLA, and one can assume equal or higher concentrations in Hudson River waterfowl during the years 1981-2007 based on the results of the 2008 waterfowl study. Therefore, PCB concentrations in mallards and other waterfowl from the Hudson River are expected to have historically exceeded the USFDA tolerance level for poultry.

The objective of this study was to determine if PCB concentrations in resident waterfowl exceed the tolerance levels enforced by USFDA for PCBs in poultry, and therefore determine if there is an injury as defined in 43 CFR 11.62(f)(1)(ii). According to these standards, an injury to a biological resource, waterfowl resident to the Upper Hudson downstream of the GE plants, has occurred.
FURTHER INFORMATION

Further information on the Hudson River natural resource damage assessment (NRDA) can be found at the following websites:


To add yourself to the Hudson-NRDA listserv:

1. Send a message to: requests@willamette.nos.noaa.gov
2. Write in the subject: Subscribe hudsonnrda

If you have questions about natural resource damages, please contact one of the individuals listed below:

Tom Brosnan
National Oceanic and Atmospheric Administration
1305 East West Highway SSMC4, Room 10219
Silver Spring, MD 20910
301-713-3038 x186
Tom.Brosnan@noaa.gov

Kathryn Jahn
United States Fish and Wildlife Service
3817 Luker Road
Cortland, NY 13045
607-753-9334
Kathryn_Jahn@fws.gov

Sean Madden
New York State Department of Environmental Conservation
Division of Fish, Wildlife, and Marine Resources
625 Broadway, 5th Floor
Albany, NY 12233
518-402-8977
ssmadden@gw.dec.state.ny.us


Figure 1: Study areas for the 2008 collection of waterfowl resident to the Hudson River, NY
Figure 2a: Study Area 1 sample locations (▲) for Hudson River resident mallards collected in July and August, 2008
**Injury Determination Report**

PCB Concentrations in Hudson River Resident Waterfowl

**Figure 2b  Study Area 2 collection locations (▲) for Hudson River resident mallards collected in July and August, 2008**

- GE Plants
- Rogers Island
- Thompson Island Dam
- Northumberland
Figure 2c  Study Area 3 collection locations (▲) for Hudson River resident mallards collected in July and August, 2008
Figure 2d  Study Area 4 collection locations (▲) for Hudson River resident mallards collected in July and August, 2008
FIGURE 3: MEAN TOTAL PCB CONCENTRATIONS IN MUSCLE FROM MALLARDS (JUVENILES AND ADULTS COMBINED) COLLECTED FROM FOUR STUDY AREAS ALONG THE HUDSON RIVER DURING JULY AND AUGUST, 2008

Notes: Error bars represent standard error. Different letters (a, b, c) represent significant differences from pairwise comparisons (Kolmogorov-Smirnov: p<0.05, with Bonferroni adjustment).
**Figure 4:** Mean total PCB concentrations in muscle from juvenile (J) and adult (A) mallards collected in July and August, 2008 from four study areas along the Hudson River.

Notes: Error bars represent standard error. Different letters (a, b, c) represent significant differences from pairwise comparisons (Kolmogorov-Smirnov: \( p<0.05 \), with Bonferroni adjustment).
**Figure 5:** Mean total PCB concentrations in fat from mallards (juveniles and adults combined) collected in July and August, 2008 from four study areas along the Hudson River.

Notes: Error bars represent standard error. Different letters (a, b, c) represent significant differences from pairwise comparisons (Kolmogorov-Smirnov: p<0.05, with Bonferroni adjustment).
Figure 6: Mean total PCB concentrations in fat from juvenile (J) and adult (A) mallards collected in July and August, 2008 from four study areas along the Hudson River.

Notes: Error bars represent standard error. Different letters (a, b, c) represent significant differences from pairwise comparisons (Kolmogorov-Smirnov: p<0.05, with Bonferroni adjustment).
Figure 7: PCB homolog distribution in muscle (M) and fat (F) for mallards (juveniles and adults combined) collected from four study areas (1, 2, 3, 4) along the Hudson River in July and August, 2008.

Notes: Homologs are expressed as a percentage of total PCBs and homolog number represents the number of chlorine atoms in each homolog group. Error bars represent standard error.
Figure 8: Total PCB concentrations in fat tissue from mallards collected from four study areas on the Hudson River during July and August, 2008

Notes: The vertical axis is a log scale. The dashed line represents the tolerance value for PCB in poultry (3 ppm on a fat basis) enforced by USFDA. Within each study area, the distribution of data along the x-axis does not reflect spatial location but rather is based on the chronological order of specimen collection.
8.0 APPENDICES

APPENDIX A. STANDARD OPERATING PROCEDURES FOR COLLECTING AND PROCESSING WATERFOWL

APPENDIX B. 2008 WATERFOWL COLLECTION INFORMATION

APPENDIX C. DATA QUALITY EVALUATION REPORTS 2008 WATERFOWL STUDY—PCB ANALYSIS

APPENDIX D. PCB CONCENTRATIONS IN WATERFOWL COLLECTED ALONG THE HUDSON RIVER, 1976-2000

APPENDIX E. 2008 WATERFOWL DATA SHEETS
APPENDIX A

STANDARD OPERATING PROCEDURES FOR COLLECTING AND PROCESSING WATERFOWL
Standard sample handling and preparation procedures for waterfowl

These juvenile and adult waterfowl sample handling and preparation procedures were developed specifically for the above referenced project. These egg-processing procedures were developed by the U.S. Fish and Wildlife Service and modified for the project based on consultation with the Quality Assurance Coordinator for this project. These procedures are to be implemented from the point of taking of the waterfowl specimens and until the resulting samples are delivered to a laboratory for chemical analysis. The procedures follow.

I. Juvenile and adult birds

A. Specimen handling prior to sample preparation

1. Once a specimen is taken in the field, it is to be placed immediately in an ice chest and chilled to 40º F or cooler. The identity of the location of collection for each bird must be maintained.

2. Specimen records: Separate Collection Record forms must be completed each day at each location. If cooperating hunters are involved, a separate Waterfowl Collection Record form must be maintained for each hunter. Determine and record on the Waterfowl Collection Record the specimen’s species identity, date and location of taking, sex and age. Measure and record total weight to the nearest 10 g (specimen should be dry and without attached or adhered debris prior to weighing) and record. Record in the notes column any information from any tags attached to the specimen at the time of taking (e.g., leg bands), or other unusual observations (e.g., deformities, blindness, etc.).

3. Attach a tag to the specimen having a unique identifying number. Record this number in the Specimen ID No. column of the Waterfowl Collection Record. The nomenclature for the Specimen ID. No. should be in accord with the following example:

   A1 MAL j M 01

   where the first two characters are the area of collection, i.e.,
   A1 = Area 1, the reference area, between the Feeder Dam and International Paper Company at Corinth;
   A2 = Area 2 between Fort Edward and Northumberland (or Fort Miller);
   A3 = Area 3 between Schuylerville and Mechanicville; and
   A4 = Area 4 from New Baltimore to Newburgh.

   The next three characters indicate species group, i.e.,
   MAL = mallard;
   TOP = target of opportunity.
The fourth character represents age of the bird, i.e.,
   j = juvenile;
   a = adult;
   e = egg.

The fifth character is the sex of the bird (no sex can be assigned to eggs), i.e.,
   M = male; and
   F = female.

The last two characters represent the number of the specimen within the classification
represented by the preceding characters. There are the following maximum numbers of birds
within each group of birds per location.

Mallard juveniles = 30 (regardless of sex);
Mallard adult males = 15;
Mallard adult females = 15;

Target of Opportunity (total) juveniles = 20 from between Fort Edward and
Mechanicville; and
Target of Opportunity (total) juveniles = 20 from between New Baltimore and
Newburgh.

4. Place specimen in a clear food grade plastic bag. Tie bag. Attach a manila ID tag to the
outside of bag. This tag is to have the following information: Abbreviated project name (e.g.,
HR Waterfowl Study), species identity, location, date, and specimen tag number.

5. Place field processed specimen back into cooler.

6. Complete Chain of Custody form for each day’s and location’s collection. If cooperating
hunters are involved, a separate record must be made for specimens taken by each hunter on
each day. Cooperating hunters must sign the Chain of Custody form and be made aware of
the terms of the Notice of Warranty on the reverse side of the Chain of Custody form.

7. Transport specimens to the frozen storage facility. All specimens should be in the custody
of one Department employee or their designee at all times. Freeze specimens as soon as
possible on the day of collection.

8. Make a copy of the Waterfowl Collection Record and Chain of Custody forms for
sampler’s records. The original Waterfowl Collection Record and Chain of Custody forms
are to remain with the samples during all future transactions.

B. Transport of samples to field laboratory

1. Specimens are to be retrieved from the freezer and placed into a cooler or ice chest with
ice, dry ice or ice packs to maintain the frozen state of the specimens. Close cooler/ice chest
and place a seal (e.g., packing, masking or duct tape) from one side across the lid to the other side of the container. Sign and date the seal with an indelible marker. This becomes the chain of custody seal.

2. Sign and complete each Chain of Custody form to acknowledge receipt of the samples.

3. Transport samples to the field laboratory at the Hale Creek Field Station, 182 Steele Avenue Extension, Gloversville, NY 12078. Notify Anthony Gudlewski (518-773-7318) prior to shipment.

4. The person receiving the samples at the field laboratory must check to assure that the Chain of Custody seal is unbroken. If the seal is broken, it must be noted on the Chain of Custody form in the Purpose of Transfer block. Break the seal to check the contents of each container to assure all specimens are present, and accept receipt of the samples by signing the Chain of Custody form. A copy of the signed Chain of Custody form shall be provided to the person delivering the specimens.

5. Once specimens are accepted at the field laboratory, the specimens are to be placed into locked frozen storage until sample preparation is to be conducted.

6. If desired by the field laboratory, individual unique field laboratory identification numbers should be assigned to specimens at this time. Each laboratory number will be placed in the Laboratory ID No. Column adjacent to the corresponding Specimen ID No. on the Waterfowl Collection Record form. The field laboratory ID number shall be in the following format: #### - # - H, where the first four # are consecutive numbers assigned to samples as they are received by the laboratory, the next two # are the last two digits of the year of sample receipt, and H represents Hale Creek Field Station.

C. Sample preparation

1. All sample preparation must be conducted in a clean work environment. The sample preparation surfaces and utensils must be cleaned with soap and water, rinsed with hexane, and then rinsed with distilled water between each sample. The preparer must use new latex or nitrile gloves for each sample prepared.

2. Remove specimen from freezer. Allow to thaw either completely or to cold but firm stage.

3. Tare an I-Chem jar that is appropriate for the mass of the sample to be taken.

4. Cut the skin the along midline on ventral side of specimen. Pull back skin from breast muscle. Excise observable subcutaneous fat from the skin and breast muscle and place into a solvent rinsed glass jar, which has been tared (pre-weighed). The preferred mass of subcutaneous fat desired is a minimum of 20 g. Weigh sample (by weighing jar with sample and subtracting jar weight) and record sample weight to the nearest 0.1 g. Place a teflon-lined screw cap on the I-Chem jar. Place an external adhesive label on the I-Chem jar. The label should contain both the Specimen ID No. and the Field Laboratory ID No. (if applicable), the
sample type, the initials of the person preparing the sample, and the date of sample preparation. The sample type should be indicated by a suffix ID (m = muscle; f = fat; e = egg) appended to the Specimen ID number, e.g.,

\[ A1 \text{ MAL} \text{ j M 01 f} \]

5. Excise the breast muscles. Assure the breast muscles are devoid of feathers or other debris. Place in tared I-Chem jar. The preferred mass of breast muscle is a minimum of 50 g. Weigh sample (by weighing jar with sample and subtracting jar weight) and record sample weight to the nearest 1.0 g. Place a teflon-lined screw cap on the I-Chem jar. Label the jar with the Specimen ID No. and the Field Laboratory ID No. (if applicable), the sample type, the initials of the person preparing the sample, and the date of sample preparation. The sample type should be indicated by a suffix ID (m = muscle; f = fat; e = egg) appended to the Specimen ID number, e.g.,

\[ A1 \text{ MAL} \text{ j M 01 m} \]

6. Re-freeze all samples and maintain in a frozen state at 0º F (−18º C). Re-package the remaining carcass with appropriate identification and re-freeze until the project is complete.

D. Sample transport to contract laboratory

1. Samples are to be retrieved from the freezer and placed into a cooler or ice chest with dry ice to maintain the frozen state of the specimens during shipment. Since dry ice is included, the outside of the container must be labeled to indicate the presence and amount (pounds) of dry ice contained therein.

2. Complete the contractor’s Laboratory Analysis Request form. Make a copy for laboratory files. Place completed forms into a clear plastic bag.

3. Sign and complete each Chain of Custody form to acknowledge transfer of the samples for the purpose of transport to the contract laboratory. Make copy of records for laboratory files. Place original completed records, including appropriate Waterfowl Collection Records, into the clear plastic bag with the Laboratory Analysis Request form(s), seal the bag, and place into the shipping container.

4. Close cooler/ice chest and place a seal (e.g., packing, masking or duct tape) from one side across the lid to the other side of the container. Sign and date the seal with an indelible marker. This becomes the chain of custody seal. Attach a completed shipping label on the container.

5. Transport samples to the contract laboratory. Shipments should occur by contract carrier with overnight delivery. All shipments should begin on either Monday, Tuesday or Wednesday to assure delivery prior to the weekend. Avoid deliveries during holiday periods. Samples are to be shipped to:
II. Eggs

A. Specimen handling prior to sample preparation

1. Upon collection of an egg in the field, weigh the egg to the nearest 0.1 g, record weight and all other appropriate information as indicated on the Waterfowl Collection Record. Place the egg in a hard container with sufficient padding to protect the egg. Label the container as in I. A. 3. above, and place container in a cooler for transport to Hale Creek Field Station, 182 Steele Avenue Extension, Gloversville, NY 12078. Complete Chain of Custody form.

2. Refrigerate specimens as soon as possible on the day of collection. Refrigerate eggs until opened, ideally no longer than 48 hours. Egg processing will be completed on a daily basis as much as practical. All specimens should be in the custody of one Department employee or their designee at all times.

3. Make a copy of the Waterfowl Collection Record and Chain of Custody forms for sampler’s records. The original Waterfowl Collection Record and Chain of Custody forms are to remain with the samples during all future transactions.

B. Sample preparation

1. Fill out the egg processing data sheet. Use one per egg.

2. Rinse the egg in cool water and gently use a sponge to remove any debris. Do not soak the egg.

3. Dry and weigh the whole egg.

4. Take three measurements of egg length and maximum egg width using calipers. Compute the average of the three measurements for final egg length and width measurements.

5. Measure the total egg volume by water displacement using a graduated cylinder. Fill the graduated cylinder with distilled water and note the starting volume. Immerse the egg using wire loops until the top of the egg is just under the surface of the water. Note the final volume, subtract the starting volume and the volume of the holding apparatus to determine final egg volume. Dry the egg.

6. Wearing nitrile gloves, create a catch basin out of aluminum foil by turning the edges up and securing the corners, so as to catch any egg contents should they spill. The foil can also
serve as a clean surface for processing instruments when not in use. Use a separate piece of foil for each sample.

7. Weigh the I-Chem jar with the lid and record the weight on the data sheet. Then place the jar in the center of the aluminum foil and loosen the lid.

8. Score the equator with a scalpel blade. Use a new scalpel blade for each egg. Cradle the egg in one hand, and gently score along the equator while rotating the egg. Continue to evenly score the egg until the membranes are revealed.

9. Remove the lid of the jar. Place the egg over the jar and cut through the membranes with the scalpel. Pour contents into the jar, or use the scalpel or forceps to remove any remaining contents. Note where the membranes are for future egg shell thickness measurements. Record the estimated stage of embryo development or any other notes. Remove any shell fragments from the jar using forceps. Cover the jar and record the weight to the nearest 0.01 g. Subtract the weight of the jar from the weight of the jar plus contents to get a weight of the contents.

10. Rinse egg shell halves with cool water and allow to dry for 10-30 days. Label each egg shell with the sample number as above in I. A. 3. Store shells in a labeled plastic bag or other container for future thickness measurements.

11. Place a label on the jar, which should be filled out as in item I.C.4 above, and prepare Chain of Custody forms. Freeze samples.

C. Sample transport to contract laboratory

   Same as I. D., above.

[If there are special shipping instructions, note them here.]
## HUDSON RIVER NATURAL RESOURCE TRUSTEE COUNCIL
### WATERFOWL COLLECTION RECORD

Print collector(s) names:
Signature of collector(s):

Method of collection: ____ Collection Area (1, 2, 3 or 4):

<table>
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<th>Field Lab ID No.</th>
<th>Specimen ID No.</th>
<th>Species</th>
<th>Location (UTM coordinates or attach topo map with location indicated)</th>
<th>Date collected</th>
<th>Sex</th>
<th>Age</th>
<th>Total weight (g)</th>
<th>Notes</th>
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Hudson River Natural Resource Trustee Council – Waterfowl 2008
Mallard Egg Processing Data Sheet

Processor(s): Name ___________________________ Name ___________________________
            Signature ________________________ Signature ________________________

Date Processed: ________________________________

Sample ID: _______________________________________________________________________

Egg Fresh Weight (g, from the field): __________

Egg Length (three measurements, mm): _______ , _______ , _______  Average _______

Egg Width (three measurements, mm): _______ , _______ , _______  Average _______

Egg Volume (ml): __________

Jar lot number __________________________   Balance within limits?  Yes OR No

Whole Egg Weight (g): __________

Contents weight:

    Weight of jar (g) : __________

    Weight of jar + contents (g): __________

    Weight of contents (g): __________

Membrane location: ___ with embryo   OR   ___ with eggshell

Contents condition (embryo development ¹, state of decay, etc.) and other comments:

_________________________________________________________________________________

_________________________________________________________________________________

Other comments:

_________________________________________________________________________________

_________________________________________________________________________________

¹ None, ¼, ½, ¾ , full term
HUDSON RIVER NATURAL RESOURCE TRUSTEE COUNCIL
CHAIN OF CUSTODY

I, _____________, of ___________________, collect
following item(s) on __________, 20__ from ____________________ ____________________
(Date) (Water Body)
in the vicinity of ____________________________________________________________
(Wetland, Bay or Cove, Landmark, Village, Road, etc.)

Town of ____________________, in ____________________________ County.

Item(s) ________________________________________________________________

Said item(s) were in my possession and handled according to standard procedures provided to me
prior to collection. The item(s) were placed in the custody of a representative of the New York State
Department of Environmental Conservation on __________, 20__,

FIRST RECIPIENT
I, _____________, received the above mentioned same(s) on the date specified and assigned identification number(s) __ to the
sample(s). I have recorded pertinent data for the sample(s) on the attached collection records. The sample(s) remained in
my custody until subsequently transferred, prepared or shipped at times and dates as attested to below.

_________________________  ________________
Signature                        Date

SECOND RECIPIENT (Print Name)    TIME & DATE    PURPOSE OF TRANSFER

SIGNATURE

UNIT

THIRD RECIPIENT (Print Name)    TIME & DATE    PURPOSE OF TRANSFER

SIGNATURE

UNIT

RECEIVED IN LABORATORY BY (Print Name)    TIME & DATE    REMARKS

SIGNATURE

UNIT

LOGGED IN BY (Print Name)    TIME & DATE    ACCESSION NUMBERS

SIGNATURE

UNIT
NOTICE OF WARRANTY

By signature to the chain of custody (reverse), the signator warrants that the information provided is truthful and accurate to the best of his/her ability. The signator affirms that he/she is willing to testify to those facts provided and the circumstances surrounding same. Nothing in this warranty or chain of custody negates responsibility nor liability of the signators for the truthfulness and accuracy of the statements provided.

HANDLING INSTRUCTIONS

On the day of collection, collector(s) name(s), address(es), date, geographic location of capture (attach a copy of topographic map or navigation chart), species, number kept of each species, and description of capture vicinity (proper noun, if possible) along with name of Town and County must be indicated on reverse.

Retain specimens in manila tagged plastic bags to avoid mixing. Note appropriate information on each bag tag.

Upon collection, keep specimens as cool as possible, preferably on ice. Freeze as soon as possible. If waterfowl are held more than 24 hours without freezing, they will not be retained or analyzed.

The initial recipient (either DEC or designated agent) of samples from collector(s) is responsible for obtaining and recording information on the collection record forms which will accompany the chain of custody. This person will seal the container using packing tape and writing his signature, time and date across the tape onto the container with indelible marker. Any time a seal is broken, for whatever purpose, the incident must be recorded on the Chain of Custody (reason, time and date) in the purpose of transfer block, then is resaled using new tape and rewriting signature, with time and date.

The appropriate completed Waterfowl Collection Record form(s) should be attached to this Chain of Custody form.
APPENDIX B

2008 WATERFOWL COLLECTION INFORMATION
PCB Concentrations in Hudson River Resident Waterfowl
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<td>4/16/2008</td>
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<td>F</td>
<td>J</td>
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<td>4592871</td>
<td>584463</td>
</tr>
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<td>J</td>
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<td>8/26/2008</td>
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<td>J</td>
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<td>A</td>
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<td>599996</td>
</tr>
</tbody>
</table>
APPENDIX C

DATA QUALITY EVALUATION REPORTS 2008 WATERFOWL STUDY—PCB ANALYSIS
Injury Determination Report
PCB Concentrations in Hudson River Resident Waterfowl
DATA QUALITY EVALUATION REPORT

HUDSON RIVER NATURAL RESOURCE DAMAGE ASSESSMENT

2008 WATERFOWL STUDY—PCB ANALYSIS

Prepared for:

State of New York
Department of Environmental Conservation

U.S. Department of Commerce
National Oceanic and Atmospheric Administration

U.S. Department of Interior
Fish and Wildlife Service

November 4, 2010

Version 1.1
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DATA QUALITY ASSESSMENT
Hudson River Natural Resource Damage Assessment
Waterfowl Study – PCB Analysis

1.0 INTRODUCTION

This report documents the results of a quality assurance review of data from duck tissue samples collected in support of the Hudson River Natural Damage Assessment. Two types of tissues from juvenile and adult birds were analyzed, breast muscle and dissectible subcutaneous fat. Samples were analyzed for the 209 PCB congeners, PCB homologs, moisture, and lipid content. Mallard eggs were tested for the same suite of analytes. The sample identifications with matrix type and associated laboratory analytical batch numbers (SDG) are provided in Attachment A: Sample Index.

Mallards were collected because they are the majority of the ducks on the Hudson River and are representative of other waterfowl species with a similar feeding preference, e.g., wood ducks. Sampling was directed separately to eggs, young-of-year (hatch year or juvenile) birds and to adults from four general areas of the river as follows:

- **Area 1**: A reference area upstream of South Glens Falls in the upper river between the Feeder Canal Dam and International Paper Company in Corinth;
- **Area 2**: Fort Edward to Northumberland [or Fort Miller] in the upper river;
- **Area 3**: Schuylerville to Mechanicville in the upper river;
- **Area 4**: New Baltimore to Newburgh in the lower river.

Mallard muscle and fat tissue samples were collected from all four study areas as indicated above, however, the analysis results related to this report are from samples collected in Areas 2 and 4. The samples that were not analyzed are being held in archive storage conditions under chain-of-custody at the NYSDEC Hale Creek Field Station.

Mallard ducks and duck eggs were collected and transported by a team from the New York State Department of Environmental Conservation (NYSDEC). Ducks and egg contents were dissected and/or preserved, and stored according to the Quality Assurance Project Plan and General Work Plan for Determining PCBs in Hudson River Resident Waterfowl: 2007-2008 Field Seasons, June 30, 2008.

All samples were shipped under chain of custody from NYSDEC to a temporary location for archive storage and then to the laboratory, Axys Analytical Services, Ltd. (Sidney, British Columbia, Canada). Axys stored the tissue and prepared aliquots of the samples for contaminant, moisture, and lipid analyses. Samples were stored, prepared, extracted, and analyzed by Axys laboratories using Standard Operating Procedures (SOPs) that were submitted and approved prior to sample receipt.
2.0 DATA VALIDATION PROCEDURES

Data validation was based on the quality assurance/quality control (QA/QC) criteria documented in the Analytical Quality Assurance Plan for the Hudson River Natural Resource Damage Assessment, (Version 2.0, September 1, 2005); National Functional Guidelines for Organic Data Review, (USEPA, 1999); and the following laboratory standard operating procedures (SOP):

- Axys MLA-007 Analytical Method for the Determination of PCB Aroclors, Total PCBs, Chlorinated Pesticides, PCB congeners, Toxaphene, and Chlorobenzenes (Revision 12).
- Additional cleanup, sample handling, storage, custody SOPs, as necessary.

Sample results and related QC data were received in both an electronic and hard copy format. Electronic data were verified against the hard copy data package. All data packages received a summary validation (Level III). Full validation of data was not performed for data generated under this study because PCB data recently generated by Axys Laboratories for other related studies received full validation and were found to be acceptable.

The following QC elements were reviewed for data packages undergoing summary validation:

- Analytical holding times
- Chain of custody and sample handling
- Method blank contamination (from summary forms)
- Instrument performance check/tuning (from summary forms)
- Initial and continuing calibration (from summary forms)
- Analytical accuracy: surrogates, spiked matrix samples, and reference material results (from summary forms)
- Analytical precision: laboratory duplicate samples (from summary forms)
- Internal standard areas (from summary forms)
- Reported detection limits (from sample result summaries)

This report summarizes the results of data validation as they relate to the analytical measurement quality objectives (MQO) for precision, accuracy, and completeness. The report also provides a quantitative and qualitative assessment of the data and identifies potential sources of error, uncertainty, and bias that may affect the overall usability.

Laboratory QC samples were used to assess the effects of homogenization procedures and to evaluate laboratory-derived contamination, laboratory performance, and sample matrix effects. Quality control samples included: method blanks, laboratory spiked matrix samples, laboratory duplicate samples, and reference material (RM) analyses. Surrogates or labeled analog compounds were added to each sample to further assess the affects of sample matrix on accuracy.
Data were qualified when associated QC sample results were outside the MQO or the method quality control criteria. The following definitions provide brief explanations of the qualifiers assigned to results in the data validation process:

**J Estimated:** The associated numerical value is an estimated quantity. The analyte was detected, but the reported value may not be accurate or precise. The J qualification indicates the data was outside the QC limits, but the exceedence was not sufficient to cause rejection of the data.

**U Not detected:** The analyte was analyzed for and a peak was detected but did not meet identification criteria and therefore, the reported value was not considered as a positive identification for the analyte.

**UJ Estimated/Not detected:** An analysis was performed for the compound or analyte, but it was not detected and the sample detection limit may be inaccurate or imprecise. The associated numerical result is the detection limit.

In addition to data qualifiers, reason codes were assigned to each qualifier to assist in data interpretation, as follows:

- **9** Precision (relative percent difference value) is greater than the specified control limit.
- **13** Surrogate recovery value is outside the specified control limits.
- **21** Potential false positive.

### 3.0 DATA QUALITY EVALUATION

The analytical data packages submitted by the laboratory were reviewed to determine whether the MQOs were met; all MQOs are listed in the Analytical Quality Assurance Plan (AQAP), Tables 6.1b - 6.1e. Each QC element is briefly discussed below; additional details are available in the method data validation reports, included in Attachment B.

#### 3.1 Holding Times and Sample Preparation

The analytes of concern for this study are persistent compounds, which have been found to remain stable in tissue after several years of storage. Therefore, no maximum holding time criterion was established for data validation. Samples were kept frozen by the laboratories at the required temperature of -20°C ±2°.

#### 3.2 Instrument Calibration

##### 3.2.1 Initial Calibration

The MQO for the initial calibration (ICAL) states that at a minimum, a five point calibration must be performed for all analytes. The percent relative standard deviation (%RSD) values for all analytes
should be less than 20%; however, up to 10% of the PCB congener %RSD values from low-resolution analysis may exceed 20% provided that all %RSD values are less than 30%.

All submitted ICAL data met the specified MQO, and were acceptable. No data were qualified based on ICAL outliers.

### 3.2.2 Continuing Calibration

The MQO for the continuing (or daily) calibrations (CCAL) requires the analysis of a continuing calibration standard at the beginning of each analytical sequence (or every 12 hours, whichever is more frequent). All percent difference (%D) values must be less than 20% for PCBs. However, up to 10% of the %D values for PCB congeners from low-resolution analysis may exceed the 20% criterion, provided that all %D values are less than 30%.

All submitted CCAL data met the specified MQO, and were acceptable. No data were qualified based on CCAL outliers.

### 3.3 Instrument Performance

#### 3.3.1 Instrument Performance Check (GC/MS)

Table 6.1b of the Analytical Quality Assurance Plan (AQAP, Version 2.0) requires that the LRMS-SIM instrument is tuned at the initiation of the analytical sequence and thereafter, every 12 hours. As a result of an on-site audit performed in December, 2004, the required frequency for LRMS-SIM instrument tuning decreased from every 12 hour to every 72 hours.

The laboratory provided tuning and calibration data to demonstrate that instrument performance remained consistent during the 72 hour period. No data were qualified based on instrument performance check sample results.

### 3.3 Blank Analyses

Laboratory (method) blanks were analyzed at the appropriate frequency of one per analytical batch, using aliquots of pure canola oil. To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank. If a contaminant is detected in an associated sample and the concentration is less than the action level, the results are qualified as not detected (U). No action is taken if the sample result is greater than the action level, or is not detected.

The laboratory assigned K-flags to PCB values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications, but are “estimated maximum possible concentrations”. When these occurred in the method blank, the results were considered as false positives. No action levels were established for these analytes.

All submitted blank data met the specified MQO, and were acceptable. Various target analytes were detected in the method blanks; however no action levels required qualification of sample results.
3.4 Accuracy

Accuracy is evaluated by comparison of an analytical concentration to a known (true) value. For the PCB analysis, accuracy was monitored using surrogate or labeled compounds and internal standards in each sample; and reference materials and spiked matrix samples (OPR) with each analytical batch. Each type of QC sample is discussed below.

3.4.1 Surrogate and Labeled Compound Recoveries

For the PCB analyses, surrogate or labeled analog compounds were added to each sample prior to extraction.

Labeled compounds were added to all samples, and most recovery values met the specified MQO of 25% to 150%. Seven (7) positive results and 12 reporting limits were estimated (J/UJ-13) because of labeled compound recovery outliers.

3.4.2 Internal Standard Recoveries

Internal standards were added at the instrument to all samples analyzed. All %R values met the specified MQO of 50% - 150%.

3.4.3 Reference Material Analyses

A reference material, White Perch Homogenate, was extracted and analyzed with 3 out of 4 analytical batches. The reference material extract was lost in a laboratory accident for SDG WG32760. This material was developed as a Hudson River fish reference material by NYSDEC. The results from this material were evaluated, however, data were not qualified if outliers occurred because the reference material is not certified. The inter-laboratory comparison study concentration value for total PCBs in the White Perch Homogenate was statistically derived from data reported as total Aroclors, total homolog groups, and as total congeners. The reference value for the material was determined from the results of each individual group and then an analysis of variance was used to compare the data sets and an overall total PCB concentration was generated. A summary of the White Perch Homogenate results for each SDG and average recovery values for Total PCB analysis is presented in Table 1A.

The Total PCBs recovered for the White Perch Homogenate reference material were within the acceptance limits for 2 of the 3 batches. The recovery of the Total PCBs in the third batch was slightly above the upper acceptance limit.

The percent lipids recovered for the reference material were within the acceptance limits for all three batches.

3.4.4 Ongoing Precision and Recovery Samples

Spiked aliquots of pure canola oil were analyzed as ongoing precision and recovery (OPR) samples for the organic analyses. The OPR samples were analyzed at the required frequency of one per analytical batch. The specified MQO requires recovery values (%R) be within the acceptance limits
of 50% - 150%; however, if a single analyte recovery value is outside the control limits, the laboratory is not required to re-extract the associated samples.

All submitted OPR data met the specified MQO, and were acceptable. No PCB data were qualified because of OPR recovery outliers.

**3.4.6 Detection Limits and Compound Identifications**

The laboratory determined sample detection limits by converting the minimum detectable signal to a concentration. The minimum detectable area is determined as three times the height of the detector noise, converted to a peak area using the area/height ratio of the corresponding surrogate peak. These limits are supported by regular MDL studies using low level spikes on tissue samples, following the statistical procedures outlined in the US Code of Federal Regulations (40 CFR Part 136, Appendix B).

Chromatography and mass spectral identification were monitored for all data. All reported positive results met identification criteria, with the following exception that was noted and flagged by the laboratory:

- The laboratory assigned K-flags to numerous values from mass spectroscopy analysis when a peak was detected but the ion abundance ratio did not meet qualitative criteria. These results were considered potential false positives or "estimated maximum possible concentrations" and were qualified during validation as not detected (U-21) at the reported values.

A total of 910 PCB congener values were assigned K flags by the laboratory and qualified as not detected (U-21) at elevated reporting limits because ion abundance ratio criteria were not met.

**3.5 Precision**

Precision is evaluated through replicate analyses of a sample. For the 2008 waterfowl study, laboratory duplicate samples and reference materials were analyzed with each analytical batch. No field duplicate samples were submitted. Overall, laboratory precision was judged as acceptable for all tissue sample analyses.

**3.5.1 Laboratory Duplicate Samples**

For samples with positive results greater than or equal to five times the MDL, duplicate precision was evaluated.

For PCBs and lipids, the specified MQO is \( \leq 30\% \) relative percent difference (RPD). If the MQO is not met, associated target analytes are estimated (J-9) in both the parent and duplicate samples. For lipid analysis, all associated samples are also qualified.

**3.5.1.1 PCB Congeners**

A laboratory duplicate sample was analyzed with each analytical batch. Forty (40) PCB results out of a total of 13,680 results were estimated (J-9) in the field samples due to laboratory precision
outliers. Overall, precision was acceptable for all PCB analyses. Table 2A provides a summary of RPD values for all positive PCB congener results.

4.0 SUMMARY OF DATA USABILITY AND COMPLETENESS

A total of 52 results were estimated because of laboratory accuracy and precision outliers.

A total of 910 PCB congener results were flagged “K” by the laboratory to indicate results that did not meet the quantitation criteria and were not positively identified; these results were considered potential false positives and were qualified as not detected (U-21) at the reported values.

The overall quality of the data is acceptable and the results, as qualified, are considered usable. There are no rejected data in this data set; therefore, the completeness for this data set is 100%.
### TABLE 1A: Summary of Reference Material Results – PCB Congeners and Lipids

**Reference Material:** White Perch Homogenate, Developed by NYSDEC

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Consensus Value (ng/g)</th>
<th>Uncertainty (ng/Kg)</th>
<th>Lower Limit (ng/Kg)</th>
<th>Upper Limit (ng/Kg)</th>
<th>WG32731 (ng/g)</th>
<th>WG32742/WG33162 (ng/g)</th>
<th>WG32760 (ng/g)</th>
<th>Average</th>
<th>Standard Deviation</th>
<th>Relative Standard Deviation (%RSD)</th>
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</thead>
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<tr>
<td>Total PCBs (ng/g)</td>
<td>948.5 44.5 714 1183</td>
<td>1210 1070 –</td>
<td>1040</td>
<td>1107 90.7 0.082%</td>
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<td></td>
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<td></td>
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</tr>
<tr>
<td>Lipids (%)</td>
<td>2.16 0.12 1.51 2.86</td>
<td>2.00 2.76 – 1040 2.40</td>
<td>2.39 0.380 0.159%</td>
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<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

1 - The White Perch Homogenate extract for SDG WG32760 was lost in a laboratory accident; therefore results are not available for this SDG.

Shaded cells contain values that exceed acceptance limits.

Upper Acceptance Limit = (Consensus Value X 1.2) + Uncertainty
Lower Acceptance Limit = (Consensus Value X 0.8) – Uncertainty
### TABLE 2A: PCB Congeners - Laboratory Duplicate, Summary of Relative Percent Difference

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<tr>
<th>SDG:</th>
<th>WG32731</th>
<th>WG32742 / WG33162</th>
<th>WG32760</th>
<th>WG32770</th>
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<td>A2MALAF15</td>
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<td>Analyte</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>PCB-1</td>
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<td>112.7%</td>
<td>126.1%</td>
</tr>
<tr>
<td>PCB-2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCB-3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCB-4/10</td>
<td>2.3%</td>
<td></td>
<td>7.3%</td>
<td></td>
</tr>
<tr>
<td>PCB-5/8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCB-6</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCB-7/9</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCB-11</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCB-12/13</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCB-14</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCB-15</td>
<td>4.6%</td>
<td>3.7%</td>
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<td>1.4%</td>
</tr>
<tr>
<td>PCB-16/32</td>
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</tr>
<tr>
<td>PCB-17</td>
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<td>PCB-20/21/33</td>
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</tr>
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<td>PCB-23/34</td>
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<td>PCB-24/27</td>
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<td></td>
<td></td>
</tr>
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<td>PCB-25</td>
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</tr>
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<td>PCB-26</td>
<td>1.3%</td>
<td></td>
<td>4.0%</td>
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</tr>
<tr>
<td>PCB-28</td>
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<td>0.4%</td>
<td>3.6%</td>
<td>2.6%</td>
</tr>
<tr>
<td>PCB-29</td>
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<td></td>
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<td>PCB-37</td>
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<td>3.7%</td>
<td>2.4%</td>
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<tr>
<td>PCB-38</td>
<td>26.5%</td>
<td></td>
<td></td>
<td></td>
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<td>PCB-42/59</td>
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<td>14.7%</td>
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<td>PCB-43/49</td>
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<td>7.3%</td>
<td>10.1%</td>
<td>2.4%</td>
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<tr>
<td>PCB-44</td>
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<td>8.3%</td>
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<td>11.8%</td>
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<tr>
<td>PCB-45</td>
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<tr>
<td>PCB-46</td>
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</tr>
<tr>
<td>PCB-47/48/75</td>
<td>2.9%</td>
<td>3.1%</td>
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TABLE 2A: PCB Congeners - Laboratory Duplicate, Summary of Relative Percent Difference (continued)

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# TABLE 2A: PCB Congeners - Laboratory Duplicate, Summary of Relative Percent Difference

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### TABLE 2A: PCB Congeners - Laboratory Duplicate, Summary of Relative Percent Difference

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Blank spaces indicate absence of RPD value because the analyte was not detected in the parent sample and/or duplicate, or because both reported values were less than 5 times the MDL.

The RPD control limit is 30%. RPD outliers are presented in **BOLD**.
### TABLE 2B: Lipids - Laboratory Duplicate, Summary of Relative Percent Difference

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<th>WG32731</th>
<th>WG32742 / WG33162</th>
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Blank spaces indicate absence of RPD value because the analyte was not detected in the parent sample and/or duplicate, or because both reported values were less than 5 times the MDL.

The RPD control limit is 30%. RPD outliers are presented in **BOLD**.
ATTACHMENT A

Sample Index
### SAMPLE INDEX of Matrix for PCB, Lipids, and Moisture Analyses

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## SAMPLE INDEX

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Total Number = 80

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ATTACHMENT B

Data Validation Report
DATA VALIDATION REPORT
2008 Waterfowl Study
PCB Congeners by AXYS Method MLA-007

This report documents the review of the data from the analysis of avian tissue and associated laboratory quality control (QC) samples. The samples were analyzed by AXYS Analytical Services, Ltd., Sidney, British Columbia, Canada. Samples were analyzed for 209 PCB congeners by AXYS Method MLA-007. Refer to the Sample Index for a complete list of samples.

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<th>No. of Samples</th>
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<td>Summary</td>
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<td>20 Fat Tissue</td>
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<tr>
<td>WG33162</td>
<td>1 Muscle Tissue</td>
<td>Summary</td>
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</table>

I. DATA PACKAGE COMPLETENESS

The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative. The laboratory submitted all required deliverables, with the following exception.

II. EDD TO HARDCOPY VERIFICATION

Sample results and related quality control data were received in both an electronic and hard copy format. Electronic data were verified against the laboratory report; no errors were found.

III. TECHNICAL DATA VALIDATION

The QC elements that were reviewed are listed below.

1. Holding Times and Sample Receipt
2. Laboratory Duplicates
   - Spiked Matrix (OPR)
1. GC/MS Instrument Performance Check
   - Initial Calibration (ICAL)
   - Continuing Calibration Verification (CVER)
2. Reference Material (RM)
2. Compound Identification and Quantitation
   - Internal Standards
   - Calculation Verification (full validation only)

1 Quality control results are discussed below, but no data were qualified.
2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

Holding Times and Sample Receipt

The analytes of concern for this study are persistent compounds, which have been found to remain stable in tissue after several years of storage; therefore, no maximum holding time criterion has been
established. AXYS maintained the samples at the required temperature of 
\(-20^\circ C \pm 2^\circ\) prior to homogenization and analysis.

**GC/MS Instrument Performance Check**

Analytical Quality Assurance Plan, Table 6.1b, (AQAP, Version 2.0) states that the GCMS instrument should be tuned at the initiation of the analytical sequence and thereafter, every 12 hours. The laboratory requested a 72 hour frequency, and provided tuning and calibration data to demonstrate that instrument performance remained consistent for periods exceeding 72 hours. This documentation was verified during an on-site audit performed in December, 2004. As a result, the required frequency for LRMS-SIM instrument tuning increased from every 12 hours to every 72 hours.

**Laboratory Blanks**

In order to assess the impact of blank contamination on the reported sample results, action levels at five times the blank concentrations are established. If the concentrations in the associated field samples are less than the action levels, the results are qualified as not detected (U-7).

The laboratory assigned K-flags to PCB values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications, but are “estimated maximum possible concentrations”. When these occurred in the method blank the results were considered as false positives. No action levels were established for these analytes.

Method blanks were analyzed at the appropriate frequency. Contaminant levels, associated samples, and action levels are documented in the data validation worksheets. Various target analytes were detected in the method blanks; however no results were qualified as not detected.

**Labeled Compound Recovery**

_SDG WG32731:_ The percent recovery (%R) values were less than the lower control limit of 25% for labeled compounds PCB202 and PCB209 in Sample A2MALJF23. The positive results and reporting limits for the associated PCB congeners were estimated (J/UJ-13) in this sample.
Laboratory Duplicates

One sample from each analytical batch was extracted and analyzed in duplicate. Where the result was > 5x the method detection limit (MDL), relative percent difference (RPD) values were calculated.

<table>
<thead>
<tr>
<th>Analytical Batch</th>
<th>Duplicate Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>WG32731</td>
<td>A2MALMJ03</td>
</tr>
<tr>
<td>WG32742</td>
<td>A2MALAM03</td>
</tr>
<tr>
<td>WG32760</td>
<td>A4MALE02</td>
</tr>
<tr>
<td>WG32770</td>
<td>A2MALAF15</td>
</tr>
</tbody>
</table>

All RPD values were less than the 30% control limit, with the following exceptions:

**SDG WG32731:** The RPD values for PCB81, PCB113, and PCB141 and total monochlorobiphenyls were greater than the control limit. The results for these analytes were estimated (J/UJ-9) in the parent and duplicate Sample A2MALJM03.

**SDG WG32742:** The RPD values for PCB1 and PCB70 were greater than the control limit. The results for these analytes were estimated (J/UJ-9) in the parent and duplicate Sample A2MALAM03.

**SDG WG32760:** The RPD values for 12 congeners and total octochlorobiphenyls were greater than the control limit, ranging from 31.4% to 112.7%. The results for these analytes were estimated (J-9) in the parent and duplicate Sample A4MALE02.

**SDG WG32770:** The RPD value for PCB1 was greater than the control limit. The results for this analyte were estimated (J-9) in the parent and duplicate Sample A2MALAF15.

Reference Material

A reference material, White Perch Homogenate, was extracted and analyzed with most analytical batches. This material was developed as a Hudson River fish reference material by NYSDEC. The results from this material were evaluated however, data were not qualified if outliers occurred. A summary of the White Perch Homogenate results and average recovery values for Total PCB analysis is presented in the Data Quality Assessment (Table 1A).

**WG32731:** The homogenate was taken from bottle number 265.

**WG32742/WG33162:** The homogenate was taken from bottle number 1061.

**WG32760:** The homogenate was taken from bottle number 1056. This sample was lost in a laboratory accident, no reference material results were reported for this SDG.

**WG32770:** The homogenate was taken from bottle number 1057.
Compound Identification & Quantitation

The laboratory assigned K-flags to numerous PCB congener values when a peak was detected but did not meet identification criteria and therefore, the reported values were not considered as positive identifications for these analytes. These results are considered potential false positives or "estimated maximum possible concentrations" and were qualified as not detected (U-21) at the reported values.

Several samples were diluted and analyzed a second time because certain PCB congener results exceeded the calibration range of the instrument. In each case the laboratory reported only the most appropriate positive result for each PCB congener, from either the original or diluted analysis.

SDG WG32731: Six muscle tissue samples were diluted and re-analyzed.

SDG WG32742: Ten muscle tissue samples were diluted and re-analyzed.

SDG WG32760: One egg content sample and fourteen fat tissue samples were diluted and re-analyzed.

SDG WG32770: Twenty fat tissue samples were diluted and re-analyzed.

IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the labeled compound, reference material, and OPR %R values, with the exceptions noted above. Precision was acceptable, as demonstrated by laboratory duplicate RPD values, with the exception noted above.

Data were estimated because of laboratory precision outliers and labeled compound %R outliers. Data were also qualified as not detected based on potential false positive results.

All other data, as qualified, are acceptable for use.
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   TABLE 1 - Summary of Reference Material Results

   TABLES 2A & 2B - Summary of Laboratory Duplicate Relative Percent Difference

   ATTACHMENT A - Sample Index

   ATTACHMENT B - Data Validation Reports
1.0 INTRODUCTION

This report documents the results of a quality assurance review of polychlorinated biphenyl (PCB) and percent lipid data from duck tissue samples collected in support of the Hudson River Natural Resource Damage Assessment. Two types of tissues from juvenile and adult birds were analyzed, breast muscle and dissectible subcutaneous fat. Samples were analyzed for the 209 PCB congeners, PCB homologs, moisture, and lipid content. The sample identifications with matrix type and associated laboratory analytical batch numbers (SDG) are provided in Attachment A: Sample Index.

Mallards were collected because they are the majority of the ducks on the Hudson River and are representative of other waterfowl species with a similar feeding preference, e.g., wood ducks. Sampling was targeted at three groups; eggs, young-of-year (hatch year or juvenile) birds, and adult birds, from four general areas of the river as follows:

- **Area 1**: A reference area upstream of South Glens Falls in the upper river between the Feeder Canal Dam and International Paper Company in Corinth
- **Area 2**: Fort Edward to Northumberland [or Fort Miller] in the upper river
- **Area 3**: Schuylerville to Mechanicville in the upper river
- **Area 4**: New Baltimore to Newburgh in the lower river

Mallard muscle and fat tissue samples were collected from all four study areas and eggs from Areas 2 and 4. Results related to this report are from muscle and fat tissue samples collected in Areas 1, 3 and 4. Samples from Area 2 and select samples from Area 4 were previously reported in the Data Quality Evaluation Report Version 1.1, November 4, 2010.

Mallard ducks were collected and transported by a team from the New York State Department of Environmental Conservation (NYSDEC). Ducks were dissected and/or preserved, and stored according to the *Quality Assurance Project Plan and General Work Plan for Determining PCBs in Hudson River Resident Waterfowl: 2007-2008 Field Seasons*, June 30, 2008.

All samples were shipped from NYSDEC to AXYS Analytical Services, Ltd. (Sidney, British Columbia, Canada). AXYS stored the tissue frozen at -20°C and prepared aliquots of the samples for PCBs, moisture, and lipid analyses. Samples were stored, prepared, extracted, and analyzed by AXYS laboratories using Standard Operating Procedures (SOPs) that were submitted and approved prior to sample receipt.
2.0 DATA VALIDATION PROCEDURES

Data validation was based on the quality assurance/quality control (QA/QC) criteria documented in the Analytical Quality Assurance Plan for the Hudson River Natural Resource Damage Assessment, (Version 2.0, September 1, 2005); National Functional Guidelines for Organic Data Review, (USEPA, 1999); and the following laboratory standard operating procedures (SOP):

- AXYS MLA-007 Analytical Method for the Determination of PCB Aroclors, Total PCBs, Chlorinated Pesticides, PCB congeners, Toxaphene, and Chlorobenzenes (Revision 13.03)
- AXYS SLA-020 Gravimetric Lipid Determination by Weight of Extract (Revision 2)
- Additional cleanup, sample handling, storage, custody SOPs, as necessary

Sample results and related QC data were received in both an electronic format and portable document format (PDF). Electronic data were verified against the PDF data package. Two SDG received a full validation (Level IV) and the remaining data received a summary validation (Level III).

The following QC elements were reviewed for data packages undergoing summary validation:

- Analytical holding times
- Chain of custody and sample handling
- Method blank contamination (from summary forms)
- Instrument performance check/tuning (from summary forms)
- Initial and continuing calibration (from summary forms)
- Analytical accuracy: labeled compounds, spiked matrix samples, and reference material results (from summary forms)
- Analytical precision: laboratory duplicate samples (from summary forms)
- Internal standard areas (from summary forms)
- Reported detection limits (from sample result summaries)

Full validation included review of all the items listed above for summary validation, plus the following QC elements:

- Compound identification (from raw data)
- Compound quantitation, transcription and calculation checks performed at a frequency of 10% from raw data. If an error was noted, 100% of the calculations and transcriptions for that data set were verified

This report summarizes the results of data validation as they relate to the analytical measurement quality objectives (MQO) for precision, accuracy, and completeness. The report also provides a quantitative and qualitative assessment of the data and identifies potential sources of error, uncertainty, and bias that may affect the overall usability.
Laboratory QC samples were used to assess the effects of homogenization procedures and to evaluate laboratory-derived contamination, laboratory performance, and sample matrix effects. Quality control samples included: method blanks, laboratory spiked matrix samples, laboratory duplicate samples, and reference material (RM) analyses. Labeled compounds were added to each sample to further assess the affects of sample matrix on accuracy.

Data were qualified when associated QC sample results were outside the MQO or the method quality control criteria. The following definitions provide brief explanations of the qualifiers assigned to results in the data validation process:

**J** Estimated: The associated numerical value is an estimated quantity. The analyte was detected, but the reported value may not be accurate or precise. The J qualification indicates the data was outside the QC limits, but the exceedance was not sufficient to cause rejection of the data.

**U** Not detected: The analyte was analyzed for and a peak was detected but did not meet identification criteria and therefore, the reported value was not considered as a positive identification for the analyte.

**UJ** Estimated/Not detected: An analysis was performed for the compound or analyte, but it was not detected and the sample detection limit may be inaccurate or imprecise. The associated numerical result is the detection limit.

In addition to data qualifiers, reason codes were assigned to each qualifier to assist in data interpretation, as follows:

- **9** Precision (relative percent difference value) is greater than the specified control limit
- **10** Spiked matrix sample recovery value is outside the specified control limits
- **13** Labeled compound recovery value is outside the specified control limits
- **21** Potential false positive

### 3.0 DATA QUALITY EVALUATION

The analytical data packages submitted by the laboratory were reviewed to determine whether the MQOs were met; all MQOs are listed in the Analytical Quality Assurance Plan (AQAP), Tables 6.1b - 6.1e. Each QC element is briefly discussed below; additional details are available in the method data validation reports included in **Attachment B**.

#### 3.1 Holding Times and Sample Preparation

The analytes of concern for this study are persistent compounds, which have been found to remain stable in tissue after several years of storage, therefore, no maximum holding time criterion was established for data validation. Samples were kept frozen by the laboratory at the required temperature of -20°C ±2°C.
3.2 Instrument Calibration

3.2.1 Initial Calibration

The MQO for the initial calibration (ICAL) states that at a minimum, a five point calibration must be performed for all analytes. The percent relative standard deviation (%RSD) values for all analytes should be less than 20%; however, up to 10% of the PCB congener %RSD values may exceed 20% provided that all %RSD values are less than 30%.

All submitted ICAL data met the specified MQO, and were acceptable.

3.2.2 Continuing Calibration

The MQO for the continuing (or daily) calibrations (CCAL) requires the analysis of a continuing calibration standard at the beginning of each analytical sequence (or every 12 hours, whichever is more frequent). All percent difference (%D) values must be less than 20% for PCBs. However, up to 10% of the %D values for PCB congeners may exceed the 20% criterion, provided that all %D values are less than 30%.

All submitted CCAL data met the specified MQO, and were acceptable.

3.3 Instrument Performance

3.3.1 Instrument Performance Check (GC/MS)

Table 6.1b of the AQAP requires that the LRMS-SIM instrument is tuned at the initiation of the analytical sequence and thereafter, every 12 hours. As a result of an on-site audit in December 2004, the required frequency for LRMS-SIM instrument tuning decreased from every 12 hour to every 72 hours.

The laboratory provided tuning and calibration data to demonstrate that instrument performance remained consistent during the 72 hour period.

3.3 Blank Analyses

Laboratory (method) blanks were analyzed at the appropriate frequency of one per analytical batch, using aliquots of pure canola oil. To assess the impact of each blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration detected in the blank.

The laboratory assigned K-flags to PCB values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications, but are “estimated maximum possible concentrations”. When these occurred in the method blank, the results were considered as false positives. No action levels were established for these analytes.

Various target analytes were detected in the method blanks; however no data required qualification based on blank contamination.
3.4 Accuracy

Accuracy is evaluated by comparison of an analytical concentration to a known (true) value. For the PCB analyses, accuracy was monitored using labeled compounds and internal standards in each sample and reference materials and spiked matrix samples (OPR) with each analytical batch. Each type of QC sample is discussed below.

3.4.1 Labeled Compound Recoveries

For the PCB analyses, labeled compounds were added to each sample prior to extraction and most recovery values met the specified MQO of 50% to 150%. Fifty-eight (58) positive results and 982 reporting limits were estimated (J/UJ-13) based on a potential low bias indicated by labeled compound recovery outliers.

3.4.2 Internal Standard Recoveries

Internal standards were added at the instrument to all samples analyzed. All percent recovery (%R) values met the specified MQO of 50% - 150%.

3.4.3 Reference Material Analyses

A reference material, White Perch Homogenate, was extracted and analyzed with 10 out of 11 analytical batches. This material was developed as a Hudson River fish reference material by NYSDEC. The results for this reference material were evaluated; however, data were not qualified based on outliers because the reference material is not certified. The reference value for total PCBs in the White Perch Homogenate was statistically derived from an inter-laboratory comparison study. The acceptance range is 80 to 120% of the 95% confidence interval of the reference value. A summary of the White Perch Homogenate results for each SDG and average recovery values for Total PCB analysis is presented in Table 1.

The Total PCBs recovered for the White Perch Homogenate reference material were within the acceptance limits for eight of the ten batches.

The percent lipids recovered for the reference material were within the acceptance limits for all ten batches.

3.4.4 Ongoing Precision and Recovery Samples

Spiked aliquots of pure canola oil were analyzed as ongoing precision and recovery (OPR) samples for the PCB analyses. The OPR samples were analyzed at the required frequency of one per analytical batch. The specified MQO for %R are 50% - 150%.

An OPR sample was analyzed with each analytical batch, and most recovery values met the specified MQO. Twenty-five (25) positive results and 18 reporting limits were estimated (J/UJ-10) because of OPR recovery outliers.
3.4.5 Detection Limits and Compound Identifications

The laboratory determined sample detection limits by converting the minimum detectable signal to a concentration. The minimum detectable area is determined as three times the height of the detector noise, converted to a peak area using the area/height ratio of the corresponding surrogate peak. These limits are supported by regular method detection limits (MDL) studies using low level spikes on tissue samples, following the statistical procedures outlined in the US Code of Federal Regulations (40 CFR Part 136, Appendix B).

Chromatography and mass spectral identification were monitored for all data. Reported positive results met identification criteria, with the exception of K-flagged results which did not meet ion ratio criteria. These results were considered to be potential false positives or "estimated maximum possible concentrations" and were qualified during validation as not detected (U-21) at the reported concentrations.

A total of 1,192 PCB congener values were assigned K-flags by the laboratory and were qualified as not detected (U-21).

3.5 Precision

Precision is evaluated through replicate analyses of a sample. For the 2011 waterfowl analysis, laboratory duplicate samples and reference materials were analyzed with each analytical batch. No field duplicate samples were submitted. Overall, laboratory precision was determined to be acceptable.

3.5.1 Laboratory Duplicate Samples

For samples with positive results greater than or equal to five times the MDL, duplicate precision was evaluated. For PCBs and lipids, the specified MQO is ≤30% relative percent difference (RPD). If the MQO is not met, associated target analytes are estimated (J/UJ-9) in both the parent and duplicate samples.

A laboratory duplicate sample was analyzed with each analytical batch. Six (6) PCB results were estimated (J-9) in the laboratory duplicate samples due to precision outliers. Table 2A provides a summary of RPD values for all positive PCB congener results. Lipids duplicate results are summarized in Table 2B.

4.0 SUMMARY OF DATA USABILITY AND COMPLETENESS

Of the 33,024 total field sample results, 2,184 results were qualified. A total of 1,088 results were estimated because of laboratory accuracy and precision outliers. A total of 1,192 PCB congener results were qualified as not detected (U-21) at the reported values due to matrix interferences. In some cases more than one qualifier was assigned to a single result.

The overall quality of the data is acceptable and the results, as qualified, are usable. There are no rejected data in this data set; completeness is 100%.
### TABLE 1: Summary of Reference Material Results
Reference Material: White Perch Homogenate, Developed by NYSDEC

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Total PCBs (ng/g)</th>
<th>Lipids (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Consensus Value</td>
<td>948.5</td>
<td>2.16</td>
</tr>
<tr>
<td>Uncertainty</td>
<td>44.5</td>
<td>0.12</td>
</tr>
<tr>
<td>Lower Limit</td>
<td>714</td>
<td>1.51</td>
</tr>
<tr>
<td>Upper Limit</td>
<td>1183</td>
<td>2.86</td>
</tr>
<tr>
<td>WG37413</td>
<td>1120</td>
<td>2.49</td>
</tr>
<tr>
<td>WG37419</td>
<td>1120</td>
<td>2.25</td>
</tr>
<tr>
<td>WG37428</td>
<td>1120</td>
<td>2.62</td>
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<td>WG37438</td>
<td>1040</td>
<td>2.25</td>
</tr>
<tr>
<td>WG37456</td>
<td>1020</td>
<td>1.78</td>
</tr>
<tr>
<td>WG37478</td>
<td>1550</td>
<td>1.81</td>
</tr>
<tr>
<td>WG37487</td>
<td>965</td>
<td>1.94</td>
</tr>
<tr>
<td>WG37504</td>
<td>1020</td>
<td>2.43</td>
</tr>
<tr>
<td>WG37505</td>
<td>1310</td>
<td>2.09</td>
</tr>
<tr>
<td>WG37530</td>
<td>1000</td>
<td>2.2</td>
</tr>
<tr>
<td>WG37845 †</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Average</td>
<td>1126.5</td>
<td>2.186</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>178.08</td>
<td>0.284</td>
</tr>
<tr>
<td>Relative Standard Deviation (%RSD)</td>
<td>15.8%</td>
<td>13.0%</td>
</tr>
</tbody>
</table>

1 - No White Perch Homogenate was analyzed with SDG WG37845. There was insufficient homogenate available.

Outliers are presented in bold.

Upper Acceptance Limit = (Consensus Value X 1.2) + Uncertainty
Lower Acceptance Limit = (Consensus Value X 0.8) – Uncertainty
### TABLE 2A: PCBs - Summary of Laboratory Duplicate Relative Percent Difference

<table>
<thead>
<tr>
<th>SDG</th>
<th>WG37413</th>
<th>WG37419</th>
<th>WG37428</th>
<th>WG37438</th>
<th>WG37456</th>
<th>WG37478</th>
<th>WG37487</th>
<th>WG37504</th>
<th>WG37505</th>
<th>WG37530</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample ID</td>
<td>A1MALAF04</td>
<td>A1MALAM16</td>
<td>A1MALAM11</td>
<td>A1MALAM16</td>
<td>A4MALAF10</td>
<td>A4MALAF01</td>
<td>A3MALAF14</td>
<td>A3MALAF06</td>
<td>A3MALJMD1</td>
<td>A3MALAF02</td>
</tr>
<tr>
<td>Matrix</td>
<td>Muscle</td>
<td>Muscle</td>
<td>Fat</td>
<td>Fat</td>
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<td>Muscle</td>
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<tr>
<td>PCB-1</td>
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<td>PCB-3</td>
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<td>PCB-4/10</td>
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<td>PCB-5/8</td>
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<tr>
<td>PCB-6</td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>PCB-7/9</td>
<td>11.7%</td>
<td>17.5%</td>
<td>26.6%</td>
<td>15.6%</td>
<td>28.0%</td>
<td>22.2%</td>
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<tr>
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</tr>
<tr>
<td>PCB-12/13</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>19.5%</td>
</tr>
<tr>
<td>PCB-14</td>
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<td></td>
<td></td>
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<tr>
<td>PCB-15</td>
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<td>PCB-16/32</td>
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<tr>
<td>PCB-20/21/33</td>
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<td>23.9%</td>
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<td>PCB-22</td>
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<td></td>
<td></td>
<td></td>
<td>7.3%</td>
</tr>
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<td>PCB-23/34</td>
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<td></td>
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<td>9.5%</td>
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<td>PCB-24/27</td>
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<td></td>
<td></td>
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<td></td>
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<td>17.4%</td>
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<td>5.7%</td>
</tr>
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<td>PCB-26</td>
<td>5.7%</td>
<td>4.2%</td>
<td>6.2%</td>
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Data Quality Evaluation
2011 Waterfowl Analysis
Version 1.0
January 20, 2012
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## TABLE 2A: PCBs - Summary of Laboratory Duplicate Relative Percent Difference

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TABLE 2A: PCBs - Summary of Laboratory Duplicate Relative Percent Difference
(continued)

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Blank spaces indicate absence of RPD value because the analyte was not detected in the parent sample and/or duplicate, or because both reported values were less than 5 times the MDL.

The RPD control limit is 30%. RPD outliers are presented in BOLD.
## TABLE 2B: Lipids - Summary of Laboratory Duplicate Relative Percent Difference

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The RPD control limit is 30%.
ATTACHMENT A

Sample Index
## SAMPLE INDEX for PCB, Lipids, and Moisture Analyses

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Total Number = 192  96  96
ATTACHMENT B

Data Validation Report
This report documents the review of the data from the analysis of waterfowl muscle or fat tissue and associated laboratory quality control (QC) samples. The samples were analyzed by AXYS Analytical Services, Ltd., Sidney, British Columbia, Canada. Samples were analyzed for 209 PCB congeners by AXYS Method MLA-007 and percent lipids by SLA-020. Refer to the Sample Index for a complete list of samples.

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I. DATA PACKAGE COMPLETENESS

The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative. The laboratory submitted all required deliverables.

II. EDD TO HARDCOPY VERIFICATION

Sample results and related quality control data were received in both an electronic and hard copy format. Electronic data were verified against the laboratory report; no errors were found.
III. TECHNICAL DATA VALIDATION

The QC elements that were reviewed are listed below.

1. Holding Times and Sample Receipt
2. Laboratory Duplicates
1. GC/MS Instrument Performance Check
   Initial Calibration (ICAL)
   Continuing Calibration Verification (CVER)
2. Spiked Matrix (OPR)
1. Reference Material (RM)
2. Compound Identification and Quantitation
   Internal Standards
1. Laboratory Blanks
2. Labeled Compound Recovery
2. Calculation Verification (full validation only)

1. Quality control results are discussed below, but no data were qualified.
2. Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

Holding Times and Sample Receipt

The analytes of concern for this study are persistent compounds, which have been found to remain stable in tissue after several years of storage; therefore, no maximum holding time criterion has been established. AXYS maintained the samples at the required temperature of $-20^{\circ}C \pm 2^{\circ}$ prior to homogenization and analysis.

GC/MS Instrument Performance Check

Table 6.1b in the Analytical Quality Assurance Plan for the Hudson Natural Resources Damage Assessment (Version 2.0); states that the GCMS instrument should be tuned at the initiation of the analytical sequence and thereafter, every 12 hours. The laboratory requested a 72 hour frequency, and provided tuning and calibration data to demonstrate that instrument performance remained consistent for periods exceeding 72 hours. This documentation was verified during an on-site audit in December 2004. As a result, the required frequency for LRMS-SIM instrument tuning increased from every 12 hours to every 72 hours.

Laboratory Blanks

Method blanks were analyzed at the appropriate frequency of one per batch. Various target analytes were detected in the method blanks. In order to assess the impact of blank contamination on the reported sample results, action levels were established at five times the blank concentrations. All associated results were either greater than the action levels or not detected; therefore no qualification of data was necessary.

The laboratory assigned K-flags to PCB values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications, but are “estimated maximum possible concentrations”. When these occurred in the method blank the results were considered to be false positives. No action levels were established for these analytes.

Labeled Compound Recovery

The percent recovery (%R) values were less than the lower control limit of 50% for several labeled compounds in one or more samples of each SDG. The positive results and reporting limits for the associated PCB congeners were estimated (J/UJ-13) in these samples. The table below lists all samples with the labeled compound outliers which resulted in qualification of data.
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<td>J/UF-13</td>
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<td>J/UF-13</td>
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<td>J/UF-13</td>
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<td>A1MALJM22</td>
<td>PCB-3L, PCB-8L</td>
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<td>J/UF-13</td>
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Laboratory Duplicates

One sample from each analytical batch was extracted and analyzed in duplicate. Where the results were greater than 5x the method detection limit (MDL), relative percent difference (RPD) values were evaluated.

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<tr>
<th>Analytical Batch</th>
<th>Duplicate Sample</th>
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<tr>
<td>WG37413</td>
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<td>WG37428</td>
<td>A1MALAM11</td>
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<td>WG37438</td>
<td>A1MALAM16</td>
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<td>WG37456</td>
<td>A4MALAF10</td>
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<td>WG37478</td>
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<td>WG37504</td>
<td>A3MALJF06</td>
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<td>WG37505</td>
<td>A3MALJM01</td>
</tr>
<tr>
<td>WG37530</td>
<td>A3MALAF02</td>
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</table>

The RPD values were less than the 30% control limit, with the following exceptions:

**SDG WG37428:** The RPD values for PCB66/80 and total tetrachlorobiphenyls were greater than the control limit. The results for these analytes were estimated (J/UJ-9) in the parent and duplicate of Sample A1MALAM11.

**SDG WG37438:** The RPD value for PCB105/127 was greater than the control limit. The results for this analyte were estimated (J-9) in the parent and duplicate of Sample A1MALAM16.

**SDG WG37456:** The RPD values for total heptachlorobiphenyls and total polychlorobiphenyls were greater than the control limit. The results for these analytes were estimated (J-9) in the parent and duplicate of Sample A4MALAF10.

**SDG WG37504:** The RPD value for PCB31 was greater than the control limit. The results for this analyte were estimated (J-9) in the parent and duplicate of Sample A3MALJF06.

Ongoing Precision and Recovery (OPR) Samples

**SDG WG37530:** The %R value for PCB 155 was less than the 50% lower control limit. The positive results or reporting limits for this analyte were estimated (J/UJ-10) for the potential low bias.

The %R values for PCB 194 and PCB 205 were greater than the 150% upper control limit. The positive results for these analytes were estimated (J-10) for the potential high bias.

Reference Material

A reference material, White Perch Homogenate, was extracted and analyzed with most analytical batches. This material was developed as a Hudson River fish reference material by New York State Department of Environmental Conservation (NYSDEC). The results from this material were
evaluated; however data were not qualified based on recovery outliers. A summary of the White Perch Homogenate results and average recovery values for Total PCB analysis is presented in the Data Quality Evaluation (Table 1).

WG37413: The homogenate was taken from Bottle 900.

WG37419: The homogenate was taken from Bottle 903.

WG37428: The homogenate was taken from Bottle 904.

WG37438: The homogenate was taken from Bottle 905.

WG37456: The homogenate was taken from Bottle 906.

WG37478: The homogenate was taken from Bottle 907. The result for total PCBs was greater than the upper control limit.

WG37487: The homogenate was taken from Bottle 908.

WG37504: The homogenate was taken from Bottle 909.

WG37505: The homogenate was taken from Bottle 910. The result for total PCBs was greater than the upper control limit.

WG37530: The homogenate was taken from Bottle 911.

WG37845: No White Perch Homogenate was analyzed with this SDG.

Compound Identification & Quantitation

The laboratory assigned K-flags to numerous PCB congener values when a peak was detected but did not meet identification criteria; therefore the reported values were not considered as positive identifications for these analytes. These results are considered potential false positives or "estimated maximum possible concentrations" and were qualified as not detected (U-21) at the reported concentration.

Several samples were diluted and re-analyzed because certain PCB congener results exceeded the calibration range of the instrument. In each case the laboratory reported only the most appropriate positive result for each PCB congener, from either the original or diluted analysis.

Calculation Verification

WG37413 & WG37487: Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

IV. OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. With the exceptions noted above, accuracy was acceptable as demonstrated by the labeled compound,
reference material, and OPR %R values and precision was acceptable as demonstrated by laboratory
duplicate RPD values.

Data were estimated because of laboratory precision outliers, labeled compound, and OPR recovery
outliers. Data were also qualified as not detected because the ion ratio criteria for positive
identification were not met.

All data, as qualified, are acceptable for use.
APPENDIX D

PCB CONCENTRATIONS IN WATERFOWL COLLECTED ALONG THE HUDSON RIVER, 1976-2000
<table>
<thead>
<tr>
<th>Species</th>
<th>Date collected (mon/yr)</th>
<th>Location (county)</th>
<th>n</th>
<th>Sub-cutaneous fat</th>
<th>Breast muscle</th>
<th>Liver</th>
<th>Brain</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>American merganser</td>
<td>12/1981</td>
<td>Ulster</td>
<td>1</td>
<td>124</td>
<td>6.3</td>
<td>na</td>
<td>na</td>
<td>na</td>
</tr>
<tr>
<td>Black duck</td>
<td>10/1979</td>
<td>Columbia</td>
<td>6</td>
<td>8.17±6.71 2.7-19</td>
<td>0.15±0.072 0.06-0.27</td>
<td>0.73 0.45-1.0 (2)</td>
<td>&lt;0.010 (2)</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>10/1981</td>
<td>Columbia</td>
<td>9</td>
<td>5.81±3.74 0.59-12</td>
<td>0.23±0.18 0.05-0.54 (8)</td>
<td>na</td>
<td>na</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>11-12/1981</td>
<td>Ulster</td>
<td>9</td>
<td>7.58±7.12 0.61-20 (8)</td>
<td>0.26±0.17 0.11-0.69</td>
<td>0.23 0.16-0.29 (2)</td>
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<tr>
<td>Fall 1983 + 1984</td>
<td>Hudson-Champlain corridor</td>
<td>11</td>
<td>7.8±13.4 &lt;0.1-43 (10)</td>
<td>0.11±0.14 &lt;0.1-0.5</td>
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<td>na</td>
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<tr>
<td>Blue-winged teal</td>
<td>10/1979</td>
<td>Columbia</td>
<td>3</td>
<td>11.4±4.98 6.1-16</td>
<td>0.78±0.44 0.32-1.2</td>
<td>0.78±0.51 0.28-1.3</td>
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<td></td>
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<td>0.07</td>
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<td>Fall 1983 + 1984</td>
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<td>3.1±7.9 0.2-28.0</td>
<td>0.09±0.11 &lt;0.1-0.4 (10)</td>
<td>na</td>
<td>na</td>
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<tr>
<td>Canada goose</td>
<td>Prior to 1976</td>
<td>Albany (presumed Hudson River)</td>
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<td>&lt;0.5</td>
<td>&lt;0.5</td>
<td>4</td>
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<td>n(^1)</td>
<td>PCB (µg/g wet weight) in:</td>
<td>Ref.(^2)</td>
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<td></td>
<td></td>
<td>Sub-cutaneous fat</td>
<td>Breast muscle</td>
<td>Liver</td>
<td>Brain</td>
<td>Other</td>
</tr>
<tr>
<td>Canvasback</td>
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<td>Ulster</td>
<td>5</td>
<td>4.32±4.94 0.98-13</td>
<td>0.26±0.27 0.11-0.66 (4)</td>
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<td>na</td>
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<tr>
<td>Fall 1983 + 1984</td>
<td>Hudson-Champlain corridor</td>
<td>14</td>
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<td>0.2±0.2 &lt;0.1-0.6 (13)</td>
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<td>Gadwall</td>
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<td>Columbia</td>
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<td>0.12</td>
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<td>Columbia</td>
<td>3(^4)</td>
<td>0.60 0.51-0.69 (2)</td>
<td>0.26 0.22-0.29 (2)</td>
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<td>0.52</td>
<td>0.05-0.98 (2)</td>
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<tr>
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<td>10/1981</td>
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<td>May 7, 1998</td>
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<td>Mallard</td>
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<td>Columbia</td>
<td>15</td>
<td>6.69±7.63 0.01-26</td>
<td>0.40±0.23 0.05-0.79</td>
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<td>0.05-1.6 (12)</td>
<td>0.46±0.78</td>
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<td>Columbia</td>
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<td>0.31±0.27 0.07-1.1 (12)</td>
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<td>0.12±0.10 &lt;0.1-0.3</td>
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\(^1\) n is the number of samples analyzed.

\(^2\) Ref. indicates the reference number for the data.

\(^3\) The sample size for this sample is 2.

\(^4\) The sample size for this sample is 1.

\(^5\) The sample size for this sample is 13.
<table>
<thead>
<tr>
<th>Species</th>
<th>Date collected (mon/yr)</th>
<th>Location (county)</th>
<th>n(^1)</th>
<th>PCB (µg/g wet weight) in:</th>
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<tr>
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<td>Sub-cutaneous fat</td>
<td>Breast muscle</td>
<td>Liver</td>
</tr>
<tr>
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<td>May 12, 1995</td>
<td>Saratoga National Historic Park</td>
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<tr>
<td>December 19, 2000</td>
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<td>4 3.42 ± 2.97 1.43 - 7.83</td>
<td>0.075 ± 0.041 0.041 - 0.129</td>
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<tr>
<td>Scaup</td>
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<td>2.7±2.4 1.0-5.5</td>
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<td>6 4.1±4.8 0.5-9.3 (5)</td>
<td>0.14±0.11 &lt;0.1-0.3</td>
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<td>Shoveller</td>
<td>10/1981</td>
<td>Columbia</td>
<td>1 8.8</td>
<td>0.30</td>
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<td>Surf scoter</td>
<td>10/1979</td>
<td>Columbia</td>
<td>1 14</td>
<td>0.71</td>
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<td>Columbia</td>
<td>2 8.2 5.4-11</td>
<td>0.58 0.22-0.94</td>
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<td>Wood duck</td>
<td>10/1981</td>
<td>Columbia</td>
<td>4 3.14±3.93 0.64-9.0</td>
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<tr>
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<td>Washington</td>
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<td>na</td>
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<tr>
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<td>Species</td>
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<td></td>
<td>Brain</td>
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<td>Other</td>
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<td>Griffin Island (Saratoga)</td>
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<td>May 7, 1998</td>
<td>Griffin Island (Saratoga)</td>
<td>2/1</td>
<td>na</td>
<td>na</td>
</tr>
</tbody>
</table>

¹ n = number of samples analyzed unless otherwise indicated in parenthesis in tissue columns. x/y = number of birds/number of samples (composite samples).

² References from which data were obtained are: 1 = Kim *et al.* (1985); 2 = Kim *et al.* (1984); 3 = O’Keefe *et al.* (2006); 4 = Baker *et al.* (1976); 5 = Foley (1992); 6 = USGS (2001a); 7 = USGS (1996); 8 = (USGS 2001b); 9 = NYSDEC unpublished data.

³ na = No analyses.

⁴ Three green-winged teal were taken but only two differing birds analyzed for each tissue.

⁵ Thirteen mallards were taken but differing numbers of birds were analyzed for each tissue.
APPENDIX E

2008 WATERFOWL DATA SHEETS
<table>
<thead>
<tr>
<th>SAMPLINGDATE</th>
<th>FIELDID</th>
<th>Matrix</th>
<th>LABORATORYID</th>
<th>SDG</th>
<th>ANALYTE</th>
<th>Value</th>
<th>InterpretiveQualifier</th>
<th>DetectionLimit</th>
<th>Units</th>
<th>WetWeight</th>
</tr>
</thead>
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<tr>
<td>7/24/2008</td>
<td>A1MALAF01</td>
<td>FAT</td>
<td>L16742-1</td>
<td>WG37428</td>
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*Qualifiers*: J = Just AOAC, U = Meets AOAC

*Detection Limits*: Given in units of wet weight (ng/g).
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