United States Department of the Interior

U. S. GEOLOGICAL SURVEY

Columbia Environmental Research Center
4200 New Haven Road
Columbia, Missouri 65201

September 14, 2000

Anne Secord
US Fish and Wildlife Service
New York Field Office
3817 Luker Road
Cortland, New York 13045

Dear Anne:

Report for Set #1: Dioxins, Furans, and non-*ortho*-PCBs in Bald Eagle Bloods is enclosed. This is the final set of data for the Eagle Bloods. The OC-pesticides, congener-specific PCBs, and the percent lipids were reported earlier in Set #1 Reports (June 30 and July 20, 2000). Please note that I titled these earlier reports as "Reports #1".

The remaining analyses for the project ("Chemical Contamination of Nesting Tree Swallows,Great Blue Herons, and Resident/Nesting Bald Eagles Along the Hudson River, New York") are in progress.

I have not yet sent you any of the Hudson River reports electronically. I can do so whenever you would like them. Drop me an email or give me a call if you have any questions about the various reports that you have received to date (573-876-1823).

Sincerely,

Carl E. Orazio
Leader, Organic Chemistry Section

USGS
science for a changing world

Columbia Environmental Research Center
U.S. Geological Survey- Biological Resources Division
4200 New Haven Road, Columbia, Missouri 65201

September 15, 2000

REPORT for Set #1
Dioxins, Furans, and non-*ortho*-PCBs in Bald Eagle Bloods

FY-00-31-02
FWS NO: 1448-50181-99-H-007
CERC NO: 3307-70L1D

By

Organic Chemistry Section
John Meadows, Robert Gale, Mike Tanner, Kevin Feltz, Paul Peterman, Kathy Echols
Carl Orazio- USGS Project Leader

FWS PROJECT TITLE

Chemical Contamination of Nesting Tree Swallows, Great Blue Herons, and Resident/Nesting Bald Eagles Along the Hudson River, New York

Principal Investigator

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Project History:

The Hudson River is highly contaminated with PCBs from industrial sources, primarily two capacitor manufacturing facilities operated by General Electric. The 200 river miles from the New York Harbor upstream to Hudson Falls, New York, are designated a Superfund Site. From 1946 until 1977, it is estimated that between 209,000 and 1.3 million pounds of PCBs were discharged into the waters of the Hudson by these two plants. Downstream movement of the PCBs was retarded by the Ft. Edward Dam until its removal in 1973, at which time the heavily contaminated sediments and detritus began to migrate downstream. In addition to contamination of the river itself, dredging operations have deposited contaminated material at nine known upland sites adjacent to the river. In 1993, it was discovered that one of the facilities was continuing to discharge PCBs into the river.

Contamination of water, sediments, and fish along the Hudson River by PCBs has been examined, but less is known about the concentration and movement of the contaminants among other trophic levels. Many resident and migrating avian species may be affected, including a fairly substantial population of wintering bald eagles (*Haliaeetus leucocephalus*). The samples described in this report are part of a series of studies designed to expand the knowledge of PCB flux in the food chain of bird species and other biota on and around the Hudson River. In 1995-1997, we took part in a study involving tree swallows (*Tachycineta bicolor*) as the indicator species along the river. Eggs, pre-fledgling chicks, odonates (emergent insects which comprise a large percentage of the diet of the swallows), and two species of ducks were assessed for contaminant concentrations. In 1997-1998, the scope of the study expanded to include samples from a bald eagle and a number of bald eagle prey species. Several species of fish, tree swallows, bluebirds, wood ducks, and two species of sparrow were analyzed.

The present segment of the study expands the diversity of the sample matrices still further. In response to the growing number of wintering, and in some cases, nesting bald eagles on the Hudson, tissue and eggs from a larger group of bald eagles and prey species have been added. The great blue heron (*Ardea herodias*), another top predator inhabiting the area, was examined. To gain further understanding of the factors influencing the life cycles and reproduction of these animals, more comprehensive organic analyses were conducted. PCB congeners including non-*ortho*-chlorinated (dioxin-like) PCBs, polychlorinated dibenzo-dioxins and-furans (PCDDs, PCDFs), polycyclic aromatic hydrocarbons (PAHs), and a suite of organochlorine pesticides were targeted in this investigation. As the information base on this ecosystem grows, a clearer picture of the remedial efforts required to restore it to its normal function will hopefully emerge.

Biota sampled by US F&WS were analyzed by the Organic Chemistry Section of the Columbia Environmental Research Center. A total of 124 samples were investigated, targeting selected analytes from the following list (each sample was not analyzed for all analytes):

Total PCBs and selected PCB congeners,
Organochlorine pesticides
2,3,7,8-substituted polychlorinated
dibenzo-*p*-dioxins (PCDDs) and –dibenzofurans (PCDDFs)
Non-*ortho* PCB congeners
Polycyclic aromatic hydrocarbons

Samples were generally grouped by analysis type. The various groups are reported separately. In addition to organic analysis, selected samples were analyzed for mercury, arsenic, and selenium; these are reported under a separate cover.

This report concerns the following samples and targeted contaminants:
19 Bald Eagle blood samples
2,3,7,8-substituted PCDD/PCDFs
non-*ortho*-PCBs

(The eagle bloods were also analyzed for congener-specific PCBs and organochlorine pesticides. These were reported on July 20, 2000).

Contents:

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I. Summary of Analytical Methods for Sample Preparation

The 19 Bald Eagle blood (serum) samples in this set were analyzed for PCDD/PCDFs and non-*ortho*-PCBs. The samples were received in two groups and were assigned CERC database numbers 19848 - 19860 and 20026 - 20031. Where serum and cells had been separated, serum was analyzed. Whole blood was analyzed as received.

Quality Control: Procedural blanks, and matrix blanks and spikes prepared from clean bovine serum were analyzed with each set of samples.

2 procedural blanks
2 bovine serum blanks
2 bovine serum spikes

Sample Preparation: The samples were dehydrated by addition of anhydrous sodium sulfate and method recovery compounds were added. Samples were extracted with methylene chloride, and a small portion of the extract (1%) was used to determine percent lipid (1). (Percent lipids were presented in the July 20th report). The remaining extracts were passed through gravity driven gel permeation chromatography (2) and High Performance Gel Permeation Chromatography (HPGPC) (3) before fractionation on a two-layered octadecyl silica/activated silica gel column into two fractions: one fraction containing PCDDs, PCDFs, PCBs and four of the targeted OCs (SODS-1), and a second fraction containing the remainder of the OCs (SODS-2) (4). SODS-2 was analyzed at this point for organochlorine pesticides by GC/ECD. SODS-1 was further fractionated on high performance Porous Graphitic Carbon (PGC) (5) into the following fractions:

PGC 1 *ortho*-chlorinated PCB congeners and four of the targeted OCs
- Analysis by gas chromatography (GC)/ electron-capture detection (ECD)

PGC 2 non-*ortho*-chlorinated PCBs
- Analysis by GC/ high resolution mass spectrometry (GC/HRMS)

PGC 3 PCDD/PCDFs
- Analysis by GC/ high resolution mass spectrometry (GC/HRMS)

II. 2,3,7,8-Cl Substituted PCDD/PCDF Analysis and Results

PCDD/PCDF fractions from PGC (PGC-3) were eluted through basic alumina for removal of potential co-contaminants such as chlorinated diphenyl ethers and residual PCNs and PCBs (6). A total of 1 ng of the internal standard, ^{13}C-labeled 1,2,3,4-TCDD, was added to each semiconical autosampler vial prior to transferring the PCDDs/PCDFs. The final extract was concentrated to a volume of ~25 µL under a stream of nitrogen. PCDFs and PCDDs were determined by GC/HRMS by monitoring five sequential mass windows of selected ions during the chromatographic separation (7).

Instrumentation:
GC/HRMS analysis was performed using a HP 5890A capillary gas chromatograph interfaced to a VG 70-AS high resolution mass spectrometer. An HP 7673 autosampler was used to introduce 2 of 25 µL of the extract from a conical vial through a spiral uniliner onto a 5 m x 320 µm deactivated fused silica retention gap via a heated (285°C) direct inlet. The analytes of interest were separated on a 50 m x 200 µm x 0.11 µm Ultra-2 (Hewlett Packard) capillary column with an initial hold of 1 min at 120 °C followed by a ramp to 200 °C at 20 °C/min, another ramp to 300 °C at 2.3 °C/min, and a final hold of 5 min. The helium carrier gas was maintained at 44 psig with an initial linear velocity of 25 cm/s. All column-to-column connections were made using fused silica press-tight connectors.

General Detection Procedure:
The VG GC/HRMS system was tuned to 10,000 R.P. and calibrated using perfluorokerosene, and mass windows were established for five ion groups to measure Cl_{4-8} PCDFs and PCDDs. These windows were monitored sequentially during the temperature program. Within each mass window, two most abundant ions were measured for positive identification and quantitation of each analyte. The ion responses were quantified and averaged, unless interferences occurred. Within each mass window, additional ions monitored any responses from potentially interfering Cl_{5-9}-PCDEs and Cl_{5-7}-PCTs, and dioxin-like Cl_{6-7}-PCNs, Cl_{3-8} dibenzothiophenes (PCDTs), and Cl_{3-8} phenanthrene and anthracenes.

Quantitation of Analytes using the Method of Isotope Dilution:
A calibration curve describing the response of each native congener to that of a ^{13}C-labeled surrogates congener was used directly in the calculations and its range of values were determined in the calibration procedure. Each calibration curve was matched to the range of analyte responses in the sample set.

Chromatographic and Mass Spectral Resolution:
Window switching times were established using a window-defining PCDF/PCDD standard mixture; relative retention times were then established for PCDTs. Chromatographic columns were selected and temperature programmed on the basis that they must resolve 2,3,7,8-TCDD from 1,2,3,7/1,2,3,8-TCDD (and from 1,2,3,4-TCDD) by a resolution factor of at least 0.5. Column performance was verified by analyzing standards of individual components, and observing the chromatographic resolution of the TCDDs, HxCDDs, and HxCDFs. Similarly, relative retention times for all other congeners of interest were evaluated with respect to labeled analogs.

Adequate mass resolution was verified while monitoring ions for Cl_{6-7} PCNs vs. ion responses of ^{13}C-TCDDs and of native TCDD vs. ^{13}C-TCDF throughout the sample set. The latter two ions, both at nominal m/z 320, differ by 0.04 Da, requiring a Resolving Power of at least 8000 for complete resolution. Monitoring these ion ratios thereby assures a continual check on mass resolution. For each mass window, lock-mass and

lock-mass-check ions were used to maintain and verify the accuracy of mass measurement.

Criteria for Confirmation:
For the positive identification and quantitation of a particular congener, the following additional criteria had to be met:

1. The peak areas for the selected ion responses must be greater than three times the background noise (S/N > 3)
2. For congeners with isotopically-labeled analogs, the ion peaks for the native must occur at retention times from -1 to +3 sec that for the corresponding ^{13}C-labeled ion peaks, which elute about 1 sec earlier than the native ion peaks;
3. For OCDF (without an isotopically-labeled analog), ion responses in sample analyses must occur at RRTs from -0.2 to 0.5% of ^{13}C-labeled OCDD, analogous to the window above;
4. For the two principal ion responses, the ion ratio must be within the acceptable range (generally ±15%) These ion ratios were determined experimentally for the system during calibrations, compared with the theoretical values, and were tracked for quality assurance.

Calculation of method efficiency (recovery of ^{13}C-surrogates): A known amount of internal standard was spiked into the final extract and used to calculate the amounts of the surrogate recovered in the final extract. The efficiency of the extraction and cleanup was measured by comparing the quantity of the surrogates detected in the final isolated extract (at GC/HRMS analysis) with the quantity spiked into the sample at the beginning of the extraction step.

Quality Control Results: In the quality control blanks, amounts of PCDFs and PCDDs are expressed as total mass (pg) divided by 3g to normalize to sample concentrations (Table 1). In these blanks, values are at or below the lowest concentrations in the samples. Concentrations of PCDFs and PCDDs in the bovine serum matrix blanks were less than the method detection limits, and approximately the same as concentrations in the procedural blanks. Concentrations of native PCDFs and PCDDs in the spiked bovine serum samples were within 25% of expected values. No positive control reference material is currently available for use for the determination of PCDFs and PCDDs in serum.

Recoveries of most of the ^{13}C-labeled surrogates (Table 2) are within the expected QC range of *25-125* %. Ion ratios of the primary ions for all detected analytes in both samples and calibration standards varied within the QC range (±15% of theoretical) except where noted by LQ. Values designated as LQ are less than the method quantification limit.

III. Non-*ortho*-PCB Congener Analysis and Results

The non-*ortho*-PCB fractions (PGC-2) were transferred to conical autosampler vials, evaporated to less than 50 µL with nitrogen, and then spiked with 5 ng of internal standard (50 µL of 100 pg/µL ^{13}C-labeled 2,2',4,5,5'-PeCB (PCB #101) in nonane). The final volume was adjusted to about 50 µL with nitrogen blow-down. Non-*ortho*-PCBs were determined by gas chromatography/high resolution mass spectrometry (GC/HRMS), monitoring two sequential mass windows of selected ions during the chromatographic separation (8,9).

Instrumentation:
GC/HRMS analysis was performed with a HP 5890A capillary gas chromatograph interfaced to a VG 70-250S high-resolution mass spectrometer. An HP 7673 autosampler was used to introduce 2 µL of the extract from a conical vial onto a 5 m x 320 µm deactivated fused silica retention gap via heated (285 °C) direct on-column injection with a Restek spiral Uniliner. A 50 m x 200 µm x 0.11 µm Ultra-1 capillary column was used to resolve non-*ortho*-PCBs from most interferents. The GC oven was held at 120 °C for 1 min, programmed to 240 °C at 2.2 °C/min, then ramped to 310 °C at 5 °C/min, and a final hold of 5 min. Helium carrier gas was maintained at 45 psig with an initial linear velocity of 27 cm/s. The analytical column was put into the MS interface, heated at 310 °C. All column-to-column connections were made with fused silica press-tight connectors.

General Detection Procedure:
The VG GC/HRMS system was tuned to 10,000 R.P. and calibrated using perfluorodecalin, and mass windows were established for two groups of non-*ortho*-PCBs. Group 1 from 23-47:00 min included ions for Cl_4-biphenyls #77 and 81 and Cl_5-biphenyl #126; Group 2 from 47:05-64 min included ions for Cl_6-biphenyl #169. Within each mass window, two most abundant ions were measured for positive identification and quantitation of each analyte. The ion responses were quantified and averaged, unless interferences occurred. Within each mass window, additional ions monitored the responses of higher chlorinated, potential interfering PCB congeners, Cl_{4-8} naphthalenes (PCNs), Cl_{3-5} terphenyls (PCTs), Br_5- and Cl_6-diphenyl ethers (residual carryover from PGC-1), and Cl_4-PCDF (to ensure no breakthrough of PCDFs).

Quantitation of Analytes:
With isotope dilution MS quantitation, the amount of each analyte detected is inherently corrected to account for losses through the whole analysis (isolation of analytes and instrumental analysis) because ^{13}C-isotopically labeled surrogates added at the beginning are recovered or lost in the same percentage as the native target analytes. A calibration curve describing the response of each native congener to that of its ^{13}C-labeled surrogate was used directly in the calculations and its range of values were determined in the calibration procedure. Each calibration curve was specifically matched to the range of analyte responses in the sample set. Concentrations of the native PCB congeners in standards ranged from 0.25 to 2,500 pg/µL.

Chromatographic and Mass Spectral Resolution:
PGC separates non-*ortho*-PCBs from other PCB congeners with nearly 99.9% efficiency. However, even this 0.1% carryover of major PCB congeners can interfere with gas chromatographic/mass spectral analysis: fragment ions are not fully resolved by high resolution MS and thus overwhelm the response of the lower level non-*ortho*-PCBs. Therefore, a 50-m Ultra 1 column is used (instead of the more commonly used DB-5 column) to chromatographically resolve most non-*ortho*-PCBs from major PCBs: non-*ortho*-Cl_4-PCB 81 elutes about 9 sec earlier than Cl_5-PCB 87, non-*ortho*-Cl_4-PCB 77 elutes about 10 sec later than Cl_6-PCB 136 and 10 sec earlier than Cl_5-PCB congener 110, and non-*ortho*-Cl_6-PCB 169 elutes when no other PCBs elute. For continuing QC checks on chromatography, molecular ion responses of these major PCB congeners are measured to ensure that their fragment ion responses do not contribute an interference >10% to the responses of the respective non-*ortho*-PCB. Column performance is verified by analyzing standards of individual congeners, labeled congeners, and congeners from Aroclor spiked mixtures.

Because non-*ortho*-Cl_5-PCB 126 is only minimally resolved from Cl_6-PCB 129, PCB 129's molecular ion response is monitored to assure that its fragment ion response (3.5% abundance) does not contribute an interference of >10% to the response of PCB 126. PCB 129's molecular ion response must not exceed three times that of PCB 126.

Adequate mass resolution is verified while monitoring ions for Cl_{4-8} PCNs throughout the sample set. The Cl_{5-7} PCNs ions monitored differ by about 0.1 Da from the ^{13}C-Cl_{4-6} PCB surrogates, assuring a continual check on mass resolution. For each mass window, lock-mass and lock-mass-check ions were used to maintain and verify the accuracy of mass measurement.

Criteria for Confirmation:
For the positive identification and quantitation of each congener, the following criteria were established and met in this study:

1. Peak areas for the selected ion responses must be greater than three times background noise.
2. Native ion peaks must occur at retention times from -1 to +3 sec that for the corresponding ^{13}C-labeled ion peaks, that elute about 1 sec earlier.
3. The ion ratio for the two principal ion responses must be within the acceptable range (generally ±15%). These ion ratios were determined experimentally for the system during calibrations, compared with the theoretical values, and were tracked.

Method efficiency by calculating percent recovery of ^{13}C-surrogates:
To account for variations in GC/HRMS analysis, a known internal standard amount was spiked into the final extract and used to calculate the amounts of the surrogates recovered in the final extract. The efficiency of the extraction and cleanup procedure was measured by comparing the quantity of the surrogates detected in the *final* isolated extract (at GC/HRMS analysis) with the quantity spiked into the sample.

Quality Control Results:
Total mass (pg) of native non-*ortho*-PCBs in the procedural blanks is normalized to sample size (3g in Table 3). For both procedural blanks, values are at or below the lowest concentrations in the samples. Non-*ortho*-PCB concentrations are also low in the bovine serum matrix blanks, and in the bovine serum matrix spike for PCDFs/PCDDs that may be considered another matrix blank for non-*ortho*-PCBs.

In the Aroclor-spiked bovine serum sample, the most abundant non-*ortho* congener, PCB 77, is within 20% of the historic mean determined for our mixed Aroclor spiking standard. Less abundant non-*ortho* congeners PCBs 81 and 126 in the Aroclor-spiked samples are also within 20% of their respective means. PCB 169 is too low for meaningful comparisons.

Percent recoveries of the ^{13}C-labeled surrogates (Table 4) range from 50 to 85%, and are within the QC range (25-125%) for this method. The corresponding native non-*ortho*-PCB concentrations would still have been accurate had losses occurred, because their values are self-corrected by the ^{13}C-labeled surrogates, using the isotope-dilution technique. Ion ratios of the primary ions for all detected analytes in both samples and calibration standards generally varied within the QC range (±15% of theoretical), except where noted by LQ (< method quantitation limit due to inaccurate ion ratio). Thus most concentrations associated with LQ are less precise and more approximate values just above the detection limit.

IV. Summary

Levels of PCDD/PCDFs and non-ortho-PCBs were determined in eagle bloods. This report completes the analysis of the eagle bloods. The organochlorine pesticides and congener-specific PCBs were reported June 30, and July 21, 2000. This report is part of the much larger investigation of exposure of biota to contaminants along the Hudson River, NY. Fish, birds, eagle prey items, eagle eggs, and eagle bloods are being analyzed for organochlorine pesticides, PCB congeners, non-ortho-PCBs, and PCDDs/PCDFs.

The PCDD/PCDFs and non-ortho-PCB data quality is described earlier in the report. The quality control samples show that the results fall well within QC limits. Background levels of the targeted contaminants were low in matrix and procedural blanks. Recoveries of the surrogates were within QC limits.

Total dioxin toxic equivalents (TEQs) based on avian TEFs (10) are presented in Table 5. The greatest contribution to dioxin-like toxicity was from the non-*ortho*-PCB congener numbers 77, 81, 126 and from the mono-ortho-PCB congener numbers 105 and 156. In this set of eagle bloods, PCDDs and PCDFs accounted for just 1-2% of the TEQs.

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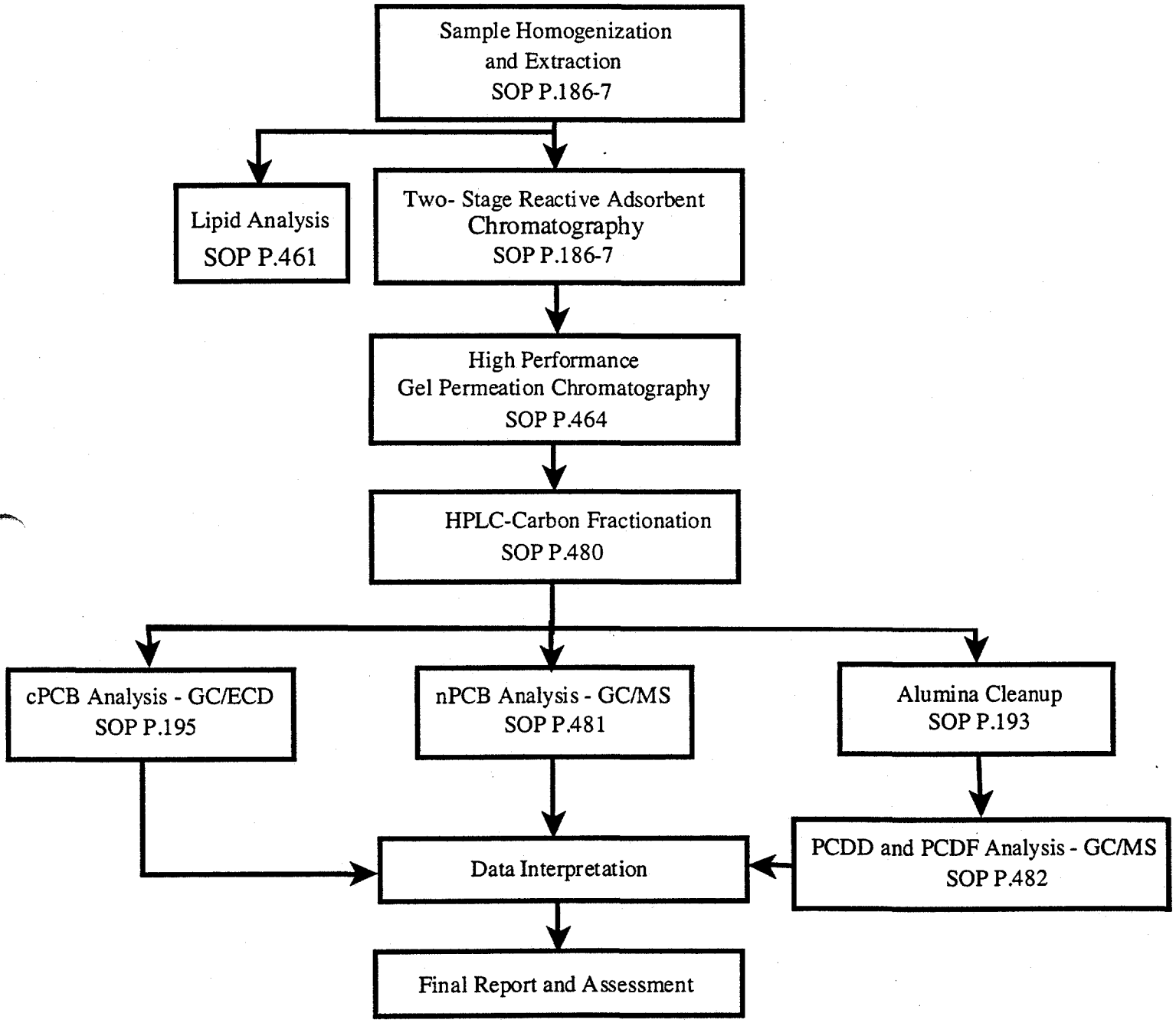
Figure 1: Analysis for Congener-specific PCB, PCDD, and PCDF Residues



Chemical Contamination of Nesting Tree Swallows, Great Blue Herons, and Resident/Nesting Bald Eagles Along the Hudson River, New York

Dioxins, Furans, and non-*ortho*-PCBs in Bald Eagle Bloods

September 14, 2000

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Table 1. 2,3,7,8-Substituted Polychlorinated Dibenzo-*p*-dioxin and Dibenzofuran Concentrations (pg/g) in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

Sample Site/Matrix:		Serum	Serum	Serum	Serum	Serum	Serum
CERC Number:		19848-S	19849-S	19850-S	19851-S	19852-S	19853-S
GC/HRMS Sets: DF33- Injection No.		33-5	33-6	33-7	33-8	33-18	33-19
Sample Submitter No.		BE-A-BL904-98	BE-A-BL949-98	BE-IM-BL935-98	BE-A-BL913-98	BE-A-BL956-98	BE-IM-BL914-98
Sample Mass Extracted (grams):		2.700	3.945	3.315	2.687	2.227	1.006
DIOXINS							
	2,3,7,8-Tetrachloro	0.2 LQ	0.2 LQ	0.7	0.6	0.3	0.2
	1,2,3,7,8-Pentachloro	0.2 LQ	0.1 ND	0.7	1.1 LQ	0.3 LQ	0.4 LQ
	1,2,3,4,7,8-Hexachloro	0.1 ND	0.1 ND	0.2 LQ	0.1 ND	0.1 ND	0.1 ND
	1,2,3,6,7,8-Hexachloro	0.1 ND	0.1 ND	0.5	0.8 LQ	0.1 LQ	0.1 LQ
	1,2,3,7,8,9-Hexachloro	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND
	1,2,3,4,6,7,8-Heptachloro	0.1	0.1 LQ	0.6	0.4	0.3 LQ	0.1 LQ
	Octachloro	18	23	67	67	24	20
FURANS	2,3,7,8-Tetrachloro	0.5 LQ	0.2 LQ	0.6 LQ	0.4 LQ	0.5 LQ	1.2 LQ
	1,2,3,7,8-Pentachloro	0.1 LQ	0.1 LQ	0.1 LQ	0.1	0.1 ND	0.2 LQ
	2,3,4,7,8-Pentachloro	0.3 LQ	0.2 LQ	1.0	0.4	0.3	0.6
	1,2,3,4,7,8-Hexachloro	0.1 ND	0.1	0.1	0.1 ND	0.1 ND	0.1 ND
	1,2,3,6,7,8-Hexachloro	0.1 LQ	0.1 LQ	0.1 LQ	0.1 LQ	0.1 ND	0.1 LQ
	1,2,3,7,8,9-Hexachloro	0.1 LQ	0.1	0.1 LQ	0.1 LQ	0.1 LQ	0.1 LQ
	2,3,4,6,7,8-Hexachloro	0.1 ND	0.1	0.1 LQ	0.1 ND	0.1 ND	0.1 LQ
	1,2,3,4,6,7,8-Heptachloro	0.1 LQ	0.1 LQ	0.1 LQ	0.1 ND	0.1 LQ	0.1
	1,2,3,4,7,8,9-Heptachloro	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND
	Octachloro	0.1 LQ	0.1 LQ	0.1	0.1 LQ	0.1	0.1 LQ

LQ Less than Method Quantification Limit due to Incomplete Ion Cluster or Ion Ratio Outside of +/- 15% Tolerances
ND Not Detected at Specified Detection Limit

Table 1. 2,3,7,8-Substituted Polychlorinated Dibenzo-*p*-dioxin and Dibenzofuran Concentrations (pg/g) in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

Sample Site/Matrix:		Serum	Serum	Serum	Serum	Serum	Serum
CERC Number:		19854-S	19855-S	19856-S	19857-S	19858-S	19859-S
GC/HRMS Sets: DF33- Injection No.		33-21	33-22	33-23	33-24	33-26	33-27
Sample Submitter No.		BE-BL921-98	BE-A-BL898-97	BE-A-BL968-99	BE-NE-BL950-98	BE-NE-BL911-A-98	BE-NE-BL911-B-98
Sample Mass Extracted (grams):		2.985	3.208	1.548	4.006	3.335	3.500
DIOXINS							
	2,3,7,8-Tetrachloro	0.5 LQ	0.9 LQ	2.7 LQ	0.1	0.1 LQ	0.1 LQ
	1,2,3,7,8-Pentachloro	0.4	0.3 LQ	1.3	0.1 LQ	0.1 LQ	0.1 LQ
	1,2,3,4,7,8-Hexachloro	0.1 ND	0.1 LQ	0.3	0.1 ND	0.1 ND	0.1
	1,2,3,6,7,8-Hexachloro	0.3	0.4	1.7	0.1 LQ	0.1 LQ	0.1 LQ
	1,2,3,7,8,9-Hexachloro	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1
	1,2,3,4,6,7,8-Heptachloro	0.3 LQ	0.2	1.1	0.1 ND	0.1 ND	0.1 LQ
	Octachloro	25	24	90	18	24	25
FURANS	2,3,7,8-Tetrachloro	0.7	0.3 LQ	1.7 LQ	2.1 LQ	0.5	0.4 LQ
	1,2,3,7,8-Pentachloro	0.2	0.1 ND	0.2	0.1 LQ	0.1 ND	0.1 LQ
	2,3,4,7,8-Pentachloro	0.3	0.7	3.5	0.2	0.2 LQ	0.1
	1,2,3,4,7,8-Hexachloro	0.1 ND	0.1 ND	0.1 LQ	0.1 LQ	0.1 ND	0.1 ND
	1,2,3,6,7,8-Hexachloro	0.1 LQ	0.1 LQ	0.1	0.1 LQ	0.1 LQ	0.1 LQ
	1,2,3,7,8,9-Hexachloro	0.1 LQ	0.1 LQ	0.1 LQ	0.1 ND	0.1 LQ	0.1 LQ
	2,3,4,6,7,8-Hexachloro	0.1 ND	0.1 LQ	0.1 ND	0.1 ND	0.1 LQ	0.1 LQ
	1,2,3,4,6,7,8-Heptachloro	0.1 LQ	0.1 LQ	0.5 LQ	0.1 ND	0.1 LQ	0.1 LQ
	1,2,3,4,7,8,9-Heptachloro	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND
	Octachloro	0.1 LQ	0.1 LQ	0.1 LQ	0.1 LQ	0.1	0.1

LQ Less than Method Quantification Limit due to Incomplete Ion Cluster or Ion Ratio Outside of +/- 15% Tolerances
ND Not Detected at Specified Detection Limit

Table 1. 2,3,7,8-Substituted Polychlorinated Dibenzo-*p*-dioxin and Dibenzofuran Concentrations (pg/g) in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

Sample Site/Matrix:		Serum	Serum	Serum	Serum	Serum	Serum
CERC Number:		19860-S	20026-S	20027-W	20028-W	20029-W	20030-W
GC/HRMS Sets: DF33- Injection No.		33-28	33-29	33-31	33-32	33-33	33-34
Sample Submitter No.		BE-NE-BL911-C-98	BE-IM-BL976-99	BE-NE-BL972A-99	BE-NE-BL972B-99	BE-NE-BL971-99	BE-NE-BL974-99
Sample Mass Extracted (grams):		3.237	2.928	3.116	3.225	3.143	3.114
DIOXINS							
	2,3,7,8-Tetrachloro	0.1 LQ	0.5	0.1	0.1 LQ	0.1 LQ	0.1 LQ
	1,2,3,7,8-Pentachloro	0.1 LQ	0.6	0.2	0.1 ND	0.1 ND	0.1 ND
	1,2,3,4,7,8-Hexachloro	0.1 ND	0.1 LQ	0.1 LQ	0.1 ND	0.1 ND	0.1 ND
	1,2,3,6,7,8-Hexachloro	0.1 ND	0.7	0.1	0.1	0.1 LQ	0.1 ND
	1,2,3,7,8,9-Hexachloro	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND
	1,2,3,4,6,7,8-Heptachloro	0.2 LQ	0.4	0.1 LQ	0.2	0.3 LQ	0.2 LQ
	Octachloro	27	19	6.0	5.2	4.6	6.4
FURANS	2,3,7,8-Tetrachloro	0.4 LQ	0.8 LQ	0.4 LQ	0.3	0.8 LQ	0.2
	1,2,3,7,8-Pentachloro	0.1 LQ	0.1 LQ	0.1 LQ	0.1 LQ	0.1 LQ	0.1
	2,3,4,7,8-Pentachloro	0.1	0.5 LQ	0.1	0.1	0.2 LQ	0.1 LQ
	1,2,3,4,7,8-Hexachloro	0.1 LQ	0.1 LQ	0.1 LQ	0.1 ND	0.1 LQ	0.1 ND
	1,2,3,6,7,8-Hexachloro	0.1 LQ	0.1 LQ	0.1 LQ	0.1	0.1 LQ	0.1
	1,2,3,7,8,9-Hexachloro	0.1 LQ	0.1 LQ	0.1 LQ	0.1 LQ	0.1 LQ	0.1 LQ
	2,3,4,6,7,8-Hexachloro	0.1 LQ	0.1 ND	0.1 LQ	0.1 LQ	0.1 LQ	0.1
	1,2,3,4,6,7,8-Heptachloro	0.1 LQ	0.1	0.1 LQ	0.1 LQ	0.1 LQ	0.1 LQ
	1,2,3,4,7,8,9-Heptachloro	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND
	Octachloro	0.1	0.1	0.1	0.1	0.1 LQ	0.1 LQ

LQ Less than Method Quantification Limit due to Incomplete Ion Cluster or Ion Ratio Outside of +/- 15% Tolerances
ND Not Detected at Specified Detection Limit

Table 1. 2,3,7,8-Substituted Polychlorinated Dibenzo-*p*-dioxin and Dibenzofuran Concentrations (pg/g) in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

Sample Site/Matrix:	Serum	Blanks:		Quality Assurance Samples	
CERC Number:	20031-W	Procedure Blank	Procedure Blank	Bovine serum Matrix Blk	Bovine serum Matrix Blk
GC/HRMS Sets: DF33- Injection No.	33-36	33-13	33-14	33-16	33-17
		########	########	########	########
Sample Submitter No.	BE-IM-BL981-99	Conc. (pg/g-eq) based on sample	Conc. (pg/g-eq) based on sample		
Sample Mass Extracted (grams):	3.57	wgts 3 g	wgts 3 g	3.0	3.0
DIOXINS					
2,3,7,8-Tetrachloro	0.3 LQ	0.1 LQ	0.1 LQ	0.1 LQ	0.1 ND
1,2,3,7,8-Pentachloro	0.3 LQ	0.1 ND	0.1 ND	0.1 ND	0.1 ND
1,2,3,4,7,8-Hexachloro	0.1 LQ	0.1 ND	0.1 ND	0.1 ND	0.1 ND
1,2,3,6,7,8-Hexachloro	0.4	0.1 ND	0.1 ND	0.1 ND	0.1 ND
1,2,3,7,8,9-Hexachloro	0.1	0.1 ND	0.1 ND	0.1 ND	0.1 ND
1,2,3,4,6,7,8-Heptachloro	0.3 LQ	0.1 ND	0.1 ND	0.1 ND	0.1 LQ
Octachloro	2.6	0.6 LQ	0.2 LQ	0.1 LQ	0.1 LQ
FURANS 2,3,7,8-Tetrachloro	0.3	0.1 LQ	3.6 LQ	0.1	0.1 LQ
1,2,3,7,8-Pentachloro	0.1 LQ	0.1	0.1 ND	0.1 ND	0.1
2,3,4,7,8-Pentachloro	0.4	0.1 ND	0.1 ND	0.1 ND	0.1 LQ
1,2,3,4,7,8-Hexachloro	0.1 ND	0.1 ND	0.1 ND	0.1 ND	0.1 ND
1,2,3,6,7,8-Hexachloro	0.1 LQ	0.1 ND	0.1 ND	0.1 ND	0.1 LQ
1,2,3,7,8,9-Hexachloro	0.1	0.1 ND	0.1 ND	0.1 LQ	0.1 LQ
2,3,4,6,7,8-Hexachloro	0.1	0.1 ND	0.1 ND	0.1 LQ	0.1 LQ
1,2,3,4,6,7,8-Heptachloro	0.1 LQ	0.1 ND	0.1 LQ	0.1 LQ	0.1 LQ
1,2,3,4,7,8,9-Heptachloro	0.1 LQ	0.1 ND	0.1 ND	0.1 ND	0.1 ND
Octachloro	0.2	0.1 ND	0.1 LQ	0.1 LQ	0.1 LQ

LQ Less than Method Quantification Limit due to Incomplete Ion Cluster or Ion Ratio Outside of +/- 15% Tolerances
ND Not Detected at Specified Detection Limit

Table 1. 2,3,7,8-Substituted Polychlorinated Dibenzo-*p*-dioxin and Dibenzofuran Concentrations (pg/g) in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

Sample Site/Matrix:	Spikes:	
CERC Number:	Bovine serum Matrix Spk	Bovine serum Matrix Spk
GC/HRMS Sets: DF33- Injection No.	33-11	33-12
	########	########
Sample Submitter No.	50 or 250 pg total	50 or 250 pg total
	(17 or 85 pg/g)	(16.6 or 82.8 pg/g)
Sample Mass Extracted (grams):	2.93	3.02
DIOXINS		
2,3,7,8-Tetrachloro	12	11
1,2,3,7,8-Pentachloro	10	10
1,2,3,4,7,8-Hexachloro	12	12
1,2,3,6,7,8-Hexachloro	11	11
1,2,3,7,8,9-Hexachloro	11	12
1,2,3,4,6,7,8-Heptachloro	11	11
Octachloro	92	81
FURANS 2,3,7,8-Tetrachloro	12	13
1,2,3,7,8-Pentachloro	11	11
2,3,4,7,8-Pentachloro	12	13
1,2,3,4,7,8-Hexachloro	13	12
1,2,3,6,7,8-Hexachloro	13	11
1,2,3,7,8,9-Hexachloro	13	14
2,3,4,6,7,8-Hexachloro	10 LQ	11
1,2,3,4,6,7,8-Heptachloro	13	13
1,2,3,4,7,8,9-Heptachloro	11	11
Octachloro	84	81

LQ Less than Method Quantification Limit due to Incomplete Ion Cluster or Ion Ratio Outside of +/- 15% Tolerances
ND Not Detected at Specified Detection Limit

Table 2. Percent Recovery of ^{13}C-Substituted Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

Sample Site/Matrix:	Serum	Serum	Serum	Serum	Serum	Serum
CERC Number:	19848-S	19849-S	19850-S	19851-S	19852-S	19853-S
GC/HRMS Sets: DF33- Injection No.	33-5	33-6	33-7	33-8	33-18	33-19
Sample Submitter No.	BE-A-BL904-98	BE-A-BL949-98	BE-IM-BL935-98	BE-A-BL913-98	BE-A-BL956-98	BE-IM-BL914-98
Sample Mass Extracted (grams):	2.700	3.945	3.315	2.687	2.227	1.006
DIOXINS						
2,3,7,8-Tetrachloro	59	55	57	53	49	53
1,2,3,7,8-Pentachloro	74	77	75	71	63	69
1,2,3,4,7,8-Hexachloro	70	70	77	74	63	70
1,2,3,6,7,8-Hexachloro	77	75	73	71	58	64
1,2,3,7,8,9-Hexachloro	69	68	69	66	55	61
1,2,3,4,6,7,8-Heptachloro	71	71	73	72	56	62
Octachloro	55	57	60	57	47	54
FURANS						
2,3,7,8-Tetrachloro	68	67	66	63	57	60
1,2,3,7,8-Pentachloro	73	70	75	71	63	68
2,3,4,7,8-Pentachloro	73	73	73	69	61	67
1,2,3,4,7,8-Hexachloro	68	69	72	70	60	66
1,2,3,6,7,8-Hexachloro	77	69	75	69	59	65
1,2,3,7,8,9-Hexachloro	67	57	68	67	56	57
1,2,3,4,6,7,8-Heptachloro	78	75	78	74	61	66
1,2,3,4,7,8,9-Heptachloro	69	62	69	67	57	58

Table 2. Percent Recovery of ^{13}C-Substituted Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

Sample Site/Matrix:	Serum	Serum	Serum	Serum	Serum	Serum
CERC Number:	19854-S	19855-S	19856-S	19857-S	19858-S	19859-S
GC/HRMS Sets: DF33- Injection No.	33-21	33-22	33-23	33-24	33-26	33-27
Sample Submitter No.	BE-BL921-98	BE-A-BL898-97	BE-A-BL968-99	BE-NE-BL950-98	BE-NE-BL911-A-98	BE-NE-BL911-B-98
Sample Mass Extracted (grams):	2.985	3.208	1.548	4.006	3.335	3.500
DIOXINS						
2,3,7,8-Tetrachloro	57	53	53	60	54	50
1,2,3,7,8-Pentachloro	75	70	72	77	72	69
1,2,3,4,7,8-Hexachloro	75	73	73	82	81	73
1,2,3,6,7,8-Hexachloro	68	63	68	73	71	64
1,2,3,7,8,9-Hexachloro	65	61	64	70	67	62
1,2,3,4,6,7,8-Heptachloro	67	63	69	78	75	66
Octachloro	56	55	57	63	61	60
FURANS						
2,3,7,8-Tetrachloro	66	60	62	67	61	55
1,2,3,7,8-Pentachloro	73	66	70	75	36	66
2,3,4,7,8-Pentachloro	71	67	69	73	71	66
1,2,3,4,7,8-Hexachloro	72	69	70	77	74	69
1,2,3,6,7,8-Hexachloro	69	60	65	65	55	64
1,2,3,7,8,9-Hexachloro	67	63	65	73	71	65
1,2,3,4,6,7,8-Heptachloro	72	68	72	80	77	71
1,2,3,4,7,8,9-Heptachloro	63	60	65	76	72	64

Table 2. Percent Recovery of ^{13}C-Substituted Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

Sample Site/Matrix:	Serum	Serum	Serum	Serum	Serum	Serum
CERC Number:	19860-S	20026-S	20027-W	20028-W	20029-W	20030-W
GC/HRMS Sets: DF33- Injection No.	33-28	33-29	33-31	33-32	33-33	33-34
Sample Submitter No.	BE-NE-BL911-C-98	BE-IM-BL976-99	BE-NE-BL972A-99	BE-NE-BL972B-99	BE-NE-BL971-99	BE-NE-BL974-99
Sample Mass Extracted (grams):	3.237	2.928	3.116	3.225	3.143	3.114
DIOXINS						
2,3,7,8-Tetrachloro	44	55	54	59	55	55
1,2,3,7,8-Pentachloro	66	68	75	78	76	79
1,2,3,4,7,8-Hexachloro	73	69	81	77	81	84
1,2,3,6,7,8-Hexachloro	67	59	71	72	71	74
1,2,3,7,8,9-Hexachloro	61	57	68	67	69	69
1,2,3,4,6,7,8-Heptachloro	70	67	73	71	75	77
Octachloro	57	57	58	65	63	63
FURANS						
2,3,7,8-Tetrachloro	51	66	61	69	62	69
1,2,3,7,8-Pentachloro	65	68	72	77	72	77
2,3,4,7,8-Pentachloro	65	66	71	73	71	75
1,2,3,4,7,8-Hexachloro	70	67	75	75	61	79
1,2,3,6,7,8-Hexachloro	65	62	67	71	53	72
1,2,3,7,8,9-Hexachloro	66	60	73	71	71	72
1,2,3,4,6,7,8-Heptachloro	72	65	79	75	67	79
1,2,3,4,7,8,9-Heptachloro	67	67	69	69	72	72

Table 2. Percent Recovery of ^{13}C-Substituted Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

Sample Site/Matrix:	Serum	Blanks:		Quality Assurance Samples	
CERC Number:	20031-W	Procedure Blank	Procedure Blank	Bovine serum Matrix Blk	Bovine serum Matrix Blk
GC/HRMS Sets: DF33- Injection No.	33-36	33-13	33-14	33-16	33-17
		########	########	########	########
Sample Submitter No.	BE-IM-BL981-99				
Sample Mass Extracted (grams):	3.57			3.0	3.0
DIOXINS					
2,3,7,8-Tetrachloro	55	52	50	51	55
1,2,3,7,8-Pentachloro	75	66	65	66	73
1,2,3,4,7,8-Hexachloro	79	63	62	65	71
1,2,3,6,7,8-Hexachloro	71	65	61	63	71
1,2,3,7,8,9-Hexachloro	67	57	54	59	63
1,2,3,4,6,7,8-Heptachloro	73	61	63	61	65
Octachloro	61	48	49	49	54
FURANS					
2,3,7,8-Tetrachloro	67	61	59	59	65
1,2,3,7,8-Pentachloro	73	69	67	65	73
2,3,4,7,8-Pentachloro	71	65	65	65	69
1,2,3,4,7,8-Hexachloro	74	61	58	61	69
1,2,3,6,7,8-Hexachloro	68	67	59	65	69
1,2,3,7,8,9-Hexachloro	73	55	55	57	62
1,2,3,4,6,7,8-Heptachloro	78	64	65	64	69
1,2,3,4,7,8,9-Heptachloro	71	57	57	59	60

Table 2. Percent Recovery of ^{13}C-Substituted Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans in Eagle Blood Samples from the Hudson River Area

File: DF33nye-blood.xls
1-Sep-2000
Date Analyzed: June 29-July 1, 2000

	Spikes:	
Sample Site/Matrix:	Bovine serum Matrix S	Bovine serum Matrix Spk
CERC Number:		
GC/HRMS Sets: DF33- Injection No.	33-11	33-12
	########	########
Sample Submitter No.	50 or 250 pg total	50 or 250 pg total
	(17 or 85 pg/g)	(16.6 or 82.8 pg/g)
Sample Mass Extracted (grams):	2.93	3.02
DIOXINS		
2,3,7,8-Tetrachloro	42	48
1,2,3,7,8-Pentachloro	54	61
1,2,3,4,7,8-Hexachloro	49	57
1,2,3,6,7,8-Hexachloro	53	57
1,2,3,7,8,9-Hexachloro	47	50
1,2,3,4,6,7,8-Heptachloro	49	55
Octachloro	32	35
FURANS		
2,3,7,8-Tetrachloro	52	59
1,2,3,7,8-Pentachloro	59	67
2,3,4,7,8-Pentachloro	53	60
1,2,3,4,7,8-Hexachloro	49	56
1,2,3,6,7,8-Hexachloro	54	62
1,2,3,7,8,9-Hexachloro	46	52
1,2,3,4,6,7,8-Heptachloro	57	61
1,2,3,4,7,8,9-Heptachloro	47	51

Table 3. Non-*o*-Chloro-Substituted PCBs (pg/g) in Bald Eagle Blood from the Hudson River Area, NY

7-Sep-00
N41-nye-blood.xls

GC/MS Sets: N41PCB
Dates: July 19-July 22, 2000

NFCR Number:	Field Number:	Sample Description:	GC/MS Run No.	Non-o-Polychlorinated Biphenyls			
				Tetra:		Penta:	Hexa:
				3,4,4',5-TCB (81)	3,3',4,4'-TCB (77)	3,3',4,4',5-PeCB (126)	3,3',4,4',5,5'-HxCB (169)
19848-S	BE-A-BL904-98	Bald Eagle Blood 2.7 g	41-12	25	107	87	15
19849-S	BE-A-BL949-98	Bald Eagle Blood 3.945 g	41-13	27	141	119	20
19850-S	BE-IM-BL935-98	Bald Eagle Blood 3.315 g	41-15	66	402	250	38 LQ
19851-S	BE-A-BL913-98	Bald Eagle Blood 2.687 g	41-16	75	106	250	44
19852-S	BE-A-BL956-98	Bald Eagle Blood 2.227 g	41-17	39	159	112	20
19853-S	BE-IM-BL914-98	Bald Eagle Blood 1.006 g	41-18	22 LQ	216	80	36
19854-S	BE-BL921-98	Bald Eagle Blood 2.985 g	41-20	24	300	70	12 LQ
19855-S	BE-A-BL898-97	Bald Eagle Blood 3.208 g	41-21	163	298	361	40
19856-S	BE-A-BL968-99	Bald Eagle Blood 1.548 g	41-22	527	2,380	1,100	89
19857-S	BE-NE-BL950-98	Bald Eagle Blood 4.006 g	41-23	29	224	48	5
19858-S	BE-NE-BL911-A-98	Bald Eagle Blood 3.335 g	41-25	19	158	41	6 LQ
19859-S	BE-NE-BL911-B-98	Bald Eagle Blood 3.5 g	41-26	13	110	27	5 LQ
19860-S	BE-NE-BL911-C-98	Bald Eagle Blood 3.237 g	41-27	8	81	21	4
20026-S	BE-IM-BL976-99	Bald Eagle Blood 2.928 g	41-28	13	196	121	26
20027-W	BE-NE-BL972A-99	Bald Eagle Blood 3.116 g	41-30	18	234	32	5 LQ
20028-W	BE-NE-BL972B-99	Bald Eagle Blood 3.225 g	41-31	12	144	22	8 LQ
20029-W	BE-NE-BL971-99	Bald Eagle Blood 3.143 g	41-32	9	105	20	4
20030-W	BE-NE-BL974-99	Bald Eagle Blood 3.114 g	41-33	10	140	27	5

Table 3. Non-*o*-Chloro-Substituted PCBs (pg/g) in Bald Eagle Blood from the Hudson River Area, NY

7-Sep-00
N41-nye-blood.xls

GC/MS Sets: N41PCB
Dates: July 19-July 22, 2000

NFCR Number:	Field Number:	Sample Description:	GC/MS Run No.	Non-o-Polychlorinated Biphenyls: Tetra: 3,4,4',5-TCB (81)	Tetra: 3,3',4,4'-TCB (77)	Penta: 3,3',4,4',5-PeCB (126)	Hexa: 3,3',4,4',5,5'-HxCB (169)
20031-W	BE-IM-BL981-99	Bald Eagle Blood 3.57 g	41-35	60	516	108	10
		Quality Control Samples:					
Proc. Blk 10/26/99		Procedure Blank, 10/26/99 **(3 g sample basis)**	41-5	**1 ND**	**22**	**4 LQ**	**2**
Proc. Blk 10/29/99		Procedure Blank, 10/29/99 **(3 g sample basis)**	41-6	**1 LQ**	**25**	**5 LQ**	**1 ND**
Matrix Blk 10/26/99		Bovine Serum Matrix Blank 10/26/99 3.0g	41-7	1 LQ	35	4 LQ	2 LQ
Matrix Blk 10/29/99		Bovine Serum Blank #2 10/29/99 3.0g	41-8	1 LQ	15	6	1 ND
Matrix Spike 10/26/99		Bovine Serum Matrix Spike-DFs 10/26/99 2.93g	41-10	1	11	3 LQ	2 LQ
Matrix Spike 10/29/99		Bovine Serum Matrix Spike 10/29/99 3.02g	41-11	21	502	16 LQ	2 LQ

LQ Less than Method Quantification Limit due to Incomplete Ion Cluster or Inaccurate Ion Ratio (Outside +/- 15% Tolerances)
ND Not Detected at Specified Detection Limit

Table 4. Percent Recoveries of ^{13}C-Non-*o*-Chloro-Substituted PCBs in Bald Eagle Blood from the Hudson River Area, NY

7-Sep-00 N41-nye-blood.xls		GC/MS Sets: N41PCB Dates: July 19-July 22, 2000		^{13}C-Non-*o*-Polychlorinated Biphenyls			
				Tetra:		Penta:	Hexa:
NFCR Number:	Submitter Number:	Sample Description:	GC/MS Run No.	3,4,4',5-TCB (^{13}C-PCB #81)	3,3',4,4'-TCB (^{13}C-PCB #77)	3,3',4,4',5-PeCB (^{13}C-PCB #126)	3,3',4,4',5,5'-HxCB (^{13}C-PCB #169)
19848-S	BE-A-BL904-98	Bald Eagle Blood 2.7 g	41-12	60	54	59	65
19849-S	BE-A-BL949-98	Bald Eagle Blood 3.945 g	41-13	68	54	56	62
19850-S	BE-IM-BL935-98	Bald Eagle Blood 3.315 g	41-15	70	55	60	65
19851-S	BE-A-BL913-98	Bald Eagle Blood 2.687 g	41-16	70	57	63	66
19852-S	BE-A-BL956-98	Bald Eagle Blood 2.227 g	41-17	62	49	53	54
19853-S	BE-IM-BL914-98	Bald Eagle Blood 1.006 g	41-18	63	52	55	59
19854-S	BE-BL921-98	Bald Eagle Blood 2.985 g	41-20	67	56	61	60
19855-S	BE-A-BL898-97	Bald Eagle Blood 3.208 g	41-21	78	65	67	71
19856-S	BE-A-BL968-99	Bald Eagle Blood 1.548 g	41-22	71	58	60	64
19857-S	BE-NE-BL950-98	Bald Eagle Blood 4.006 g	41-23	71	60	64	66
19858-S	BE-NE-BL911-A-98	Bald Eagle Blood 3.335 g	41-25	81	65	75	73
19859-S	BE-NE-BL911-B-98	Bald Eagle Blood 3.5 g	41-26	76	64	76	77
19860-S	BE-NE-BL911-C-98	Bald Eagle Blood 3.237 g	41-27	79	69	77	78
20026-S	BE-IM-BL976-99	Bald Eagle Blood 2.928 g	41-28	71	60	64	67
20027-W	BE-NE-BL972A-99	Bald Eagle Blood 3.116 g	41-30	79	60	66	63
20028-W	BE-NE-BL972B-99	Bald Eagle Blood 3.225 g	41-31	74	63	64	65
20029-W	BE-NE-BL971-99	Bald Eagle Blood 3.143 g	41-32	77	62	67	66
20030-W	BE-NE-BL974-99	Bald Eagle Blood 3.114 g	41-33	83	65	73	79

Table 4. Percent Recoveries of ^{13}C-Non-o-Chloro-Substituted PCBs in Bald Eagle Blood from the Hudson River Area, NY

7-Sep-00 N41-nye-blood.xls		GC/MS Sets: N41PCB Dates: July 19-July 22, 2000		^{13}C-Non-o-Polychlorinated Biphenyls			
				Tetra:		Penta:	Hexa:
NFCR Number:	Submitter Number:	Sample Description:	GC/MS Run No.	3,4,4',5-TCB (^{13}C-PCB #81)	3,3',4,4'-TCB (^{13}C-PCB #77)	3,3',4,4',5-PeCB (^{13}C-PCB #126)	3,3',4,4',5,5'-HxCB (^{13}C-PCB #169)
20031-W	BE-IM-BL981-99	Bald Eagle Blood 3.57 g	41-35	77	63	67	69
		Quality Control Samples:					
Proc. Blk 10/26/99		Procedure Blank, 10/26/99	41-5	58	50	51	55
Proc. Blk 10/29/99		Procedure Blank, 10/29/99	41-6	62	52	55	62
Matrix Blk 10/26/99		Bovine Serum Matrix Blank 10/26/99 3.0g	41-7	64	53	53	59
Matrix Blk 10/29/99		Bovine Serum Blank #2 10/29/99 3.0g	41-8	67	56	62	65
Matrix Spike 10/26/99		Bovine Serum Matrix Spike-DFs 10/26/99 2.93g	41-10	70	56	58	57
Matrix Spike 10/29/99		Bovine Serum Matrix Spike 10/29/99 3.02g	41-11	85	67	75	76

Table 5. Calculated Avian TEQs for Eagle Blood (pg/g)

Sample ID		Mono-*ortho* congeners								Sum mPCB
		123	118	114	105	167	156	157	189	
	Avian TEFs[1]	0.00001	0.00001	0.0001	0.0001	0.00001	0.0001	0.0001	0.00001	
	TEQs (pg/g)									
19848-s		0.011	0.465	0.109	1.550	0.030	0.639	0.166	0.011	3.0
19849-s		0.014	0.568	0.193	1.660	0.070	1.045	0.173	0.014	3.7
19850-s		0.028	1.076	0.452	4.024	0.066	1.267	0.318	0.017	7.2
19851-s		0.036	1.203	0.515	4.106	0.081	1.473	0.345	0.020	7.8
19852-s		0.015	0.600	0.183	2.307	0.038	0.642	0.193	0.013	4.0
19853-s		0.005	0.319	0.087	1.263	0.019	0.243	0.104	0.007	2.0
19854-s		0.006	0.258	0.088	1.045	0.019	0.220	0.086	0.006	1.7
19855-s		0.056	2.124	0.785	6.912	0.147	3.384	0.529	0.032	14
19856-s		0.195	8.923	2.925	26.023	0.450	12.175	1.859	0.103	53
19857-s		0.010	0.429	0.125	1.505	0.019	0.350	0.103	0.005	2.5
19858-s		0.007	0.288	0.091	1.070	0.015	0.246	0.078	0.005	1.8
19859-s		0.004	0.171	0.062	0.667	0.008	0.121	0.047	0.003	1.1
19860-s		0.003	0.134	0.048	0.538	0.008	0.108	0.041	0.003	0.88
20026-s		0.003	0.190	0.047	0.621	0.019	0.259	0.094	0.007	1.2
20027-w		0.005	0.187	0.069	0.705	0.011	0.152	0.055	0.003	1.2
20028-w		0.002	0.101	0.042	0.393	0.005	0.061	0.028	0.002	0.63
20029-w		0.001	0.064	0.028	0.276	0.004	0.041	0.025	0.002	0.44
20030-w		0.002	0.097	0.037	0.389	0.005	0.076	0.033	0.002	0.64
20031-w		0.007	0.323	0.123	1.219	0.013	0.262	0.075	0.004	2.0

[1]From Van den Berg etal. 1998.

Table 5. Calculated Avian TEQs for Eagle Blood (pg/g)

Sample ID		Non-*ortho* congeners 81	77	126	169	Sum nPCB
	Avian TEFs[1]	0.1	0.05	0.1	0.001	
	TEQs (pg/g)					
19848-s		2.5	5.3	8.7	0.015	16
19849-s		2.7	7.0	12	0.020	22
19850-s		6.6	20	25	0.038	52
19851-s		7.5	5.3	25	0.044	38
19852-s		3.9	7.9	11	0.020	23
19853-s		2.2	11	8.0	0.036	21
19854-s		2.4	15	7.0	0.012	24
19855-s		16	15	36	0.040	67
19856-s		53	119	110	0.089	282
19857-s		2.9	11	4.8	0.0049	19
19858-s		1.9	7.9	4.1	0.0057	14
19859-s		1.3	5.5	2.7	0.0045	9.5
19860-s		0.81	4.1	2.1	0.0035	6.9
20026-s		1.3	9.8	12	0.026	23
20027-w		1.8	12	3.2	0.0050	17
20028-w		1.2	7.2	2.2	0.0085	11
20029-w		0.94	5.2	2.0	0.0037	8.1
20030-w		0.99	7.0	2.7	0.0045	11
20031-w		6.0	26	11	0.01	43

[1]From Van den Berg etal. 1998.

Table 5. Calculated Avian TEQs for Eagle Blood (pg/g)

Sample ID		Dioxins 2378-TCDD	12378-PCDD	123478-HxCDD	123678-HxCDD	123789-HxCDD	1234678-HpCDD	OCDD	Total Dioxins
	Avian TEFs[1]	1	1	0.05	0.01	0.1	0.001	0.0001	
	TEQs (pg/g)								
19848-s		0.20	0.20	0.005	0.001	0.010	0.0001	0.0018	0.42
19849-s		0.20	0.10	0.005	0.001	0.010	0.0001	0.0023	0.32
19850-s		0.70	0.70	0.010	0.005	0.010	0.0006	0.0067	1.4
19851-s		0.60	1.1	0.005	0.008	0.010	0.0004	0.0067	1.7
19852-s		0.30	0.30	0.005	0.001	0.010	0.0003	0.0024	0.62
19853-s		0.20	0.40	0.005	0.001	0.010	0.0001	0.0020	0.62
19854-s		0.50	0.40	0.005	0.003	0.010	0.0003	0.0025	0.92
19855-s		0.90	0.30	0.005	0.004	0.010	0.0002	0.0024	1.2
19856-s		2.7	1.3	0.015	0.017	0.010	0.0011	0.0090	4.1
19857-s		0.10	0.10	0.005	0.001	0.010	0.0001	0.0018	0.22
19858-s		0.10	0.10	0.005	0.001	0.010	0.0001	0.0024	0.22
19859-s		0.10	0.10	0.005	0.001	0.010	0.0001	0.0025	0.22
19860-s		0.10	0.10	0.005	0.001	0.010	0.0002	0.0027	0.22
20026-s		0.50	0.60	0.005	0.007	0.010	0.0004	0.0019	1.1
20027-w		0.10	0.20	0.005	0.001	0.010	0.0001	0.0006	0.32
20028-w		0.10	0.10	0.005	0.001	0.010	0.0002	0.0005	0.22
20029-w		0.10	0.10	0.005	0.001	0.010	0.0003	0.0005	0.22
20030-w		0.10	0.10	0.005	0.001	0.010	0.0002	0.0006	0.22
20031-w		0.30	0.30	0.005	0.004	0.010	0.0003	0.0003	0.62

[1]From Van den Berg etal. 1998.

Table 5. Calculated Avian TEQs for Eagle Blood (pg/g)

Sample ID		Furans							
		2378-TCDF	12378-PCDF	23478-PCDF	123478-HxCDF	123678-HxCDF	123789-HxCDF	234678-HxCDF	1234678-HpCDF
	Avian TEFs[1]	1	0.1	1	0.1	0.1	0.1	0.1	0.01
	TEQs (pg/g)								
19848-s		0.50	0.01	0.30	0.01	0.01	0.01	0.01	0.00
19849-s		0.20	0.01	0.20	0.01	0.01	0.01	0.01	0.00
19850-s		0.60	0.01	1.0	0.01	0.01	0.01	0.01	0.00
19851-s		0.40	0.01	0.40	0.01	0.01	0.01	0.01	0.00
19852-s		0.50	0.01	0.30	0.01	0.01	0.01	0.01	0.00
19853-s		1.2	0.02	0.60	0.01	0.01	0.01	0.01	0.00
19854-s		0.70	0.02	0.30	0.01	0.01	0.01	0.01	0.00
19855-s		0.30	0.01	0.70	0.01	0.01	0.01	0.01	0.00
19856-s		1.7	0.02	3.5	0.01	0.01	0.01	0.01	0.01
19857-s		2.1	0.01	0.20	0.01	0.01	0.01	0.01	0.00
19858-s		0.50	0.01	0.20	0.01	0.01	0.01	0.01	0.00
19859-s		0.40	0.01	0.10	0.01	0.01	0.01	0.01	0.00
19860-s		0.40	0.01	0.10	0.01	0.01	0.01	0.01	0.00
20026-s		0.80	0.01	0.50	0.01	0.01	0.01	0.01	0.00
20027-w		0.40	0.01	0.10	0.01	0.01	0.01	0.01	0.00
20028-w		0.30	0.01	0.10	0.01	0.01	0.01	0.01	0.00
20029-w		0.80	0.01	0.20	0.01	0.01	0.01	0.01	0.00
20030-w		0.20	0.01	0.10	0.01	0.01	0.01	0.01	0.00
20031-w		0.30	0.01	0.40	0.01	0.01	0.01	0.01	0.00

[1]From Van den Berg etal. 1998.

Table 5. Calculated Avian TEQs for Eagle Blood (pg/g)

Sample ID		1234789-HpCDF	OCDF	Total Furans	Total TEQs
	Avian TEFs[1]	0.01	0.0001		
	TEQs (pg/g)				
19848-s		0.00	0.00001	0.85	21
19849-s		0.00	0.00001	0.45	26
19850-s		0.00	0.00001	1.7	62
19851-s		0.00	0.00001	0.85	48
19852-s		0.00	0.00001	0.85	29
19853-s		0.00	0.00001	1.9	26
19854-s		0.00	0.00001	1.1	28
19855-s		0.00	0.00001	1.1	84
19856-s		0.00	0.00001	5.3	344
19857-s		0.00	0.00001	2.4	24
19858-s		0.00	0.00001	0.75	17
19859-s		0.00	0.00001	0.55	11
19860-s		0.00	0.00001	0.55	8.6
20026-s		0.00	0.00001	1.4	27
20027-w		0.00	0.00001	0.55	19
20028-w		0.00	0.00001	0.45	12
20029-w		0.00	0.00001	1.1	10
20030-w		0.00	0.00001	0.35	12
20031-w		0.00	0.00002	0.75	46

[1]From Van den Berg etal. 1998.

United States Department of the Interior

U. S. GEOLOGICAL SURVEY

Columbia Environmental Research Center
4200 New Haven Road
Columbia, Missouri 65201

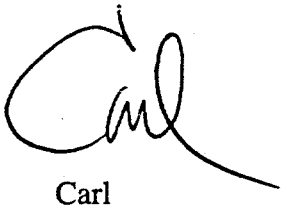
September 14, 2000

Anne Secord
US Fish and Wildlife Service
New York Field Office
3817 Luker Road
Cortland, New York 13045

Dear Anne:

While looking over the earlier report for OC pesticides and PCBs I found a text error on page 6. I am enclosing the title page of the report for reference, the page with the error shown, and the replacement page.

Thanks,



Carl

Anne-
page 6 error and
replacement page is attached.

USGS
science for a changing world

Columbia Environmental Research Center
U.S. Geological Survey- Biological Resources Division
4200 New Haven Road, Columbia, Missouri 65201

July 20, 2000

REPORT #1
PCBs and OC Pesticides in Bald Eagle Blood
FY-00-31-02
FWS NO: 1448-50181-99-H-007
CERC NO: 3307-70L1D

By

Organic Chemistry Section
John Meadows, Kathy Echols, Robert Gale, Paul Peterman
Carl Orazio- USGS Project Leader

FWS PROJECT TITLE

Chemical Contamination of Nesting Tree Swallows, Great Blue Herons, and Resident/Nesting Bald Eagles Along the Hudson River, New York

Principal Investigator

Anne Secord
US Fish and Wildlife Service
New York Field Office
3817 Luker Road
Cortland, New York 13045

Peter Nye
Endangered Species Unit
Wildlife Resources Center
New York State Department of Environmental Conservation
Delmar, New York 12054

except for procedural blank samples, which are reported as a mass amount (ng). Quality control data for procedural and matrix blanks, spikes, replicates, and positive controls are presented in Table 2. The method detection limits (MDLs) for individual PCB congeners and for total PCBs are based on procedural blank (PB) results according to the method outlined by Keith *et al.* (7,8). Briefly, an average and standard deviation are determined. The MDL (ng) is calculated using the following formula:

$$MDL = (PB\ Avg) + 3(PB\ SD).$$

The MDL is then expressed in units of concentration: mass of analyte per mass of sample. If sample masses are within 10% of each other, an average mass is calculated for the entire set. The lowest MDL for this set of samples was 0.01 ng/g and 3 ng/g for the highest (9) of the individual PCB congeners and the MDL 30 ng/g for total PCB concentrations. If included, the method quantitation limit (MQL) was calculated as well using the formula (7,8):

$$MQL = (PB\ Avg) + 10(PB\ SD).$$

The mass corrections are made in the same manner as for the MDLs. Two congeners (048, 085) were non-quantifiable due to known interferences (Table 1).

Gas chromatographic analysis, peak measurement decisions, and quantification were monitored with triplicate injection of the same sample. Precision averaged 4% for all the sample sets.

Accuracy of the method is monitored through rigorous quality control. Analytical standards have been verified against certified standards. Analyte recoveries are monitored by the following spikes:

1) internal recovery standards in each sample,
2) PCB-spiked control bovine serum.

The spiked recovery compounds, PCBs 029, 155, and 204, which elute in the PGC1 fraction, are presented in Table 3. PCB 029, a trichlorobiphenyl, is representative of more volatile early eluting PCBs (Cl_1 - Cl_3). PCB 155, a hexachlorobiphenyl, is representative of mid-range eluting congeners (Cl_4 - Cl_6). PCB 204, an octachlorobiphenyl, is less volatile and representative of later eluting PCBs (Cl_7 - Cl_{10}). Recoveries averaged 33 ± 11% for PCB 029, 52 ± 15% for PCB 155, and 61 ± 17% for PCB 204 (Table 3). ~~Recoveries of spiked A1111 PCB congeners ranged from 3% to 121% and~~ recovery of total PCBs were 69% for the matrix spike. ERROR

III. Organochlorine Pesticide Analysis and Results

Organochlorine pesticide fractions (SODS-1/PGC 1 and SODS-2) were adjusted to a final volume of 2 mL and 80 ng internal standard (PCBs 030 and 207) was added.

except for procedural blank samples, which are reported as a mass amount (ng). Quality control data for procedural and matrix blanks, spikes, replicates, and positive controls are presented in Table 2. The method detection limits (MDLs) for individual PCB congeners and for total PCBs are based on procedural blank (PB) results according to the method outlined by Keith *et al.* (7,8). Briefly, an average and standard deviation are determined. The MDL (ng) is calculated using the following formula:

$$MDL = (PB\ Avg) + 3(PB\ SD).$$

The MDL is then expressed in units of concentration: mass of analyte per mass of sample. If sample masses are within 10% of each other, an average mass is calculated for the entire set. The lowest MDL for this set of samples was 0.01 ng/g and 3 ng/g for the highest (9) of the individual PCB congeners and the MDL 30 ng/g for total PCB concentrations. If included, the method quantitat calculated as well using the formula (7,8):

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replacement page
OC & PCBs - EAGLE BLOODS
REPORT #1.
— Carl

$$MQL = (PB\ Avg) + 10(PB\ SD).$$

The mass corrections are made in the same manner as for the congeners (048, 085) were non-quantifiable due to known inte

Gas chromatographic analysis, peak measurement decisions, and quantification were monitored with triplicate injection of the same sample. Precision averaged 4% for all the sample sets.

Accuracy of the method is monitored through rigorous quality control. Analytical standards have been verified against certified standards. Analyte recoveries are monitored by the following spikes:

1) internal recovery standards in each sample,
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The spiked recovery compounds, PCBs 029, 155, and 204, which elute in the PGC1 fraction, are presented in Table 3. PCB 029, a trichlorobiphenyl, is representative of more volatile early eluting PCBs (Cl_1 - Cl_3). PCB 155, a hexachlorobiphenyl, is representative of mid-range eluting congeners (Cl_4 - Cl_6). PCB 204, an octachlorobiphenyl, is less volatile and representative of later eluting PCBs (Cl_7 - Cl_{10}). Recoveries averaged 33 ± 11% for PCB 029, 52 ± 15% for PCB 155, and 61 ± 17% for PCB 204 (Table 3). The total-PCB recovery in the matrix spike was 70%.

III. Organochlorine Pesticide Analysis and Results

Organochlorine pesticide fractions (SODS-1/PGC 1 and SODS-2) were adjusted to a final volume of 2 mL and 80 ng internal standard (PCBs 030 and 207) was added.