

United States Department of the Interior

U. S. GEOLOGICAL SURVEY

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

July 21, 2000

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

Dear Anne:

The organochlorine pesticide results for the bald eagle bloods have been added to "Report #1" of the study entitled, "Chemical Contamination of Nesting Tree Swallows, Great Blue Herons, and Resident/Nesting Bald Eagles Along the Hudson River, New York". This report updates the previous report that contained just the congener-specific PCB results. Please note that we have presented the %lipid results in a more appropriate manner than shown previously with the PCB results where the values had too many significant figures. We evaluated this measurement and because of the very low sample mass and the low percent lipid in these bloods, presenting the %lipid value as a range better reflects the precision. Please give me a call if this needs any clarification.

The PCDDs, PCDFs, and non-ortho-PCBs information for the eagle bloods is being generated: PCDD/Fs are being identified/quantified and the non-ortho fractions are currently being analyzed by GC/HRMS. A report of the PCDD/F and non-ortho information looks promising in the next three to four weeks. We are finishing the review of the congener-specific PCB and pesticide results for a large part of the tree swallow investigation (CERC #18759-18799) and for all of the Great Blue Heron brains (#18803-18818). We will be able to mail that report to you by the end of this month.

The analyses of the other Hudson River samples are still proceeding well. The prey items have been analyzed by GC/ECD and identification/quantification/reporting will soon begin. Give me a call if you have any questions. We can send the reports electronically whenever you would like. My phone number is 573-876-1823.

Sincerely,

Carl E. Orazio, Ph.D. Leader, Organic Chemistry Section



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

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 1997-1999, 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, 1999-2001, 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. Congener-specific PCBs, non-*ortho*-chlorinated (dioxin-like) PCBs, polychlorinated dibenzo-*p*-dioxins and dibenzofurans (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 Fish & Wildlife Service were analyzed by the Organic Chemistry Section of the Columbia Environmental Research Center. The following analytes were targeted: Total PCBs and selected PCB congeners,

Organochlorine pesticides,

- 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxins and -dibenzofurans, Non-*ortho* PCB congeners,
- Polycyclic aromatic hydrocarbons,

In addition to organic analysis, selected samples will be analyzed by inductively coupled plasma for mercury, arsenic, and selenium. The inorganic analysis will be reported under a separate cover.

A total of 124 samples were investigated. The samples were subdivided into smaller groups and the results of the analysis of the groups will be reported in separate reports. This, the first of those reports, concerns concentrations of organochlorine pesticides and PCB congeners in a set of 19 Bald Eagle blood samples

Contents:

- I. Summary of Analytical Methods for Sample Preparation
- II. PCB Congener (cPCB) Analysis and Results
- III. Organochlorine Pesticide Analysis and Results

IV. Summary of PCB and Pesticide Results

Tables:

- 1. PCB congener Concentrations in Eagle Blood
- 2. Eagle Blood PCBs QC Samples and Detection Limit Calculations
- 3. Procedural PCB Spike Recoveries
- 4. Organochlorine Pesticides in Eagle Blood
- 5. Organochlorine Pesticide QC—Eagle Blood
- 6. Procedural Recoveries for Organochlorine Pesticides

Figures:

1. Analytical scheme for organochlorine pesticides, congener-specific PCBs, non-*ortho*-PCBs, PCDFs, and PCDDs.

I. Summary of Analytical Methods for Sample Preparation

The 19 Bald Eagle blood (serum) samples in this set were analyzed for PCB congeners and a suite of organochlorine pesticides (OCs). 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:

Matrix QC samples (blanks and spikes) prepared from clean bovine serum were analyzed with each set of samples.

All samples, including QC samples were spiked with method recovery compounds (40 ng each) before extraction to monitor recoveries through the cleanup procedures. The following compounds were used:

PCB 029 (2,4,6-trich!orobiphenyl) PCB155 (2,2',4,4',6,6'-hexachlorobiphenyl) PCB 204 (2,2',3,4,4',5,6,6'-octachlorobiphenyl) tetra-chloro-*meta*-xylene dibutylchlorendate.

Matrix spikes also were spiked with:

Organochlorine pesticides (24 compounds; 40 ng each) PCBs (mixed Aroclors 1242, 1248, 1254, 1260; 1000 ng total Aroclors).

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). Because of limited sample size and low percent lipids the detectable level is 0.15%, and the precision varies as shown in Tables 1 and 4. 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 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 polychlorinated dibenzo-*p*-dioxins and -furans (PCDD/PCDFs) - Analysis by GC/ high resolution mass spectrometry (GC/HRMS)

PGC 1 results are reported in this document. PGC 2 and PGC 3 results will be reported later.

II. PCB Congener Analysis and Results

The sample extracts were adjusted to a final volume of 2 mL, and 80 ng of the internal standard mix (PCBs 030 and 207) was added. After vortex mixing, a portion of the solution was transferred to a labeled autosampler vial. Individual PCB congeners were measured in PGC1 fractions by GC/ECD. Results of the PCB analysis are presented in Tables 1 - 3.

<u>Instrumentation:</u> Analyses were performed as described in CERC SOP P.195 (6), a dual column method, using Hewlett-Packard 5890 Series II GCs with cool on-column capillary injection systems and Hewlett-Packard model 7673 autosamplers. For all analyses, a 3-m section of 0.53 mm i.d. uncoated and deactivated (Restek Corp., Inc.) capillary retention gap was attached to the front of each analytical column by a "Press-Tight" (Restek Corp., Inc.) union. The analytical columns were a 60-m x 0.25-mm DB-5 (0.25μ m 5%-phenyl-methylpolysiloxane, J&W Scientific) and a 60-m x 0.25-mm DB-17 (0.25μ m 50%-phenyl-methylpolysiloxane, J&W Scientific) . The H₂-carrier gas was pressure regulated at 25 psi. The temperature program for the PCB analysis was as follows: initial temperature 60°C, immediately ramped to 150°C at 15°C/min, then ramped to 250°C at 1°C/min, and finally ramped to 320°C.

General Detection and Quantification Procedure: Capillary GC/ECD data were collected, archived in digital form, and processed using a PE-Nelson chromatography data system which included the model 970 interface and version 4.1 of Turbochrom[™] chromatography software on a Pentium microcomputer. Six to nine levels of PCB standards, a combination of Aroclors 1242, 1248, 1254, 1260 in 1:1:1:1 w/w/w/w ratio (designated A1111), were used for PCB congeners calibration, with total PCB concentrations ranging from 10 to 8000 ng/mL. An instrumental internal standard (IIS) method with PCB congener 030 or 207 used to calculate the concentrations of the targeted compounds. Samples were processed and analyzed in two batches. PCB congener results are presented in Table 1, designated by their CERC database number and are cross-referenced to their field identification number, site and description. Concentrations are expressed as nanograms of analyte per gram of sample (wet weight).

<u>Quality Control Procedures and Results</u>: Recovery data for PCBs 030, 155, and 204 are presented in Table 3. All concentrations are reported in nanograms per gram,

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(P5 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.

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.

After vortex mixing, a portion of the solution was transferred to a labeled autosampler vial. Individual organochlorine pesticides were measured in both fractions by GC/ECD. Results of the OC pesticide analysis are presented in Tables 4 - 6.

Instrumentation: Analyses were performed as described in CERC SOP P.459 (10), using Hewlett-Packard 5890 Series II GCs with cool on-column capillary injection systems and Hewlett-Packard model 7673 autosamplers. For all analyses, a 3-m section of 0.53 mm i.d. uncoated and deactivated (Restek Corp., Inc.) capillary retention gap was attached to the front of the analytical column by a "Press-Tight" (Restek Corp., Inc.) union. The analytical column for the SODS-2 fraction was a 30-m x 0.25-mm DB-1 (methylsilicone, J&W Scientific). The SODS-1 fractions were analyzed on the a 60 m DB-5 in order to confirm the identification of OC pesticides. The H₂-carrier gas was pressure regulated at 11 psi. The temperature program for the analysis was as follows: initial temperature 90°C, immediately ramped to 165°C at 15°C/min, held 3 minutes, then ramped to 260°C at 2.5°C/min with a 5 minute hold, and finally ramped to 320°C at 10°C/min, and held for 1 min. The temperature of the ECD was held at 330°C.

<u>General Detection and Quantification Procedure:</u> Capillary GC/ECD data were collected, archived in digital form, and processed using a PE-Nelson chromatography data system which included the model 970 interface and version 4.1 of Turbochrom[™] chromatography software on a Pentium microcomputer. Six levels of OC pesticide standards were used for calibration, with each pesticide at concentrations ranging from 1 to 80 ng/mL. An instrumental internal standard (IIS) method with either PCB 030 or 207 was used to calculate the concentrations of the targeted compounds. Samples were analyzed and processed in two batches. Organochlorine pesticide results are presented in Table 4, designated by their CERC database number and are cross-referenced to their field identification number, site, and sample description. Concentrations are expressed as nanograms of analyte per gram of sample (wet weight).

<u>Quality Control Procedures and Results</u>: Quality control data for procedural and matrix blanks, spikes, replicates, and positive controls are presented in Table 5. All concentrations of QC samples are reported in total nanograms, except for matrix blank samples, which are reported as nanograms per gram. The method detection limits (MDLs) for individual compounds are calculated by the method described in the previous section and are mass normalized as discussed above.

The gas chromatographic precision of the pesticide analysis was determined to be 2% for this OC pesticide sample set based upon triplicate injections of the same sample (MS 102699 #1). This measures variability in gas chromatographic analysis (injection, etc.), peak measurement decisions, and quantification.

Recoveries were monitored by the analysis of spiked control bovine serum. The procedural recovery compounds, PCBs 029 and 204 elute in SODS-1. Recoveries of these compounds averaged $40 \pm 7\%$ and $71 \pm 4\%$, respectively (Table 6). SODS-1

compound concentrations, HCB, heptachlor were corrected using PCB 029 recoveries; p,p'-DDE, and mirex, were corrected using PCB 204 recoveries. The SODS-2 recovery compound, dibutylchlorendate, averaged 89 ± 8% recovery. The OC pesticides that elute in SODS-2 were corrected using dibutylchlorendate recoveries. DDT compounds averaged 87 ± 15%; chlordanes averaged 85 ± 2%; BHC compounds averaged 82 ± 6%; endosulfans averaged 85 ± 3%.

IV. Summary of PCB and OC-Pesticide Results

This report is part of the much larger investigation of exposure of biota to contaminants along the Hudson River, NY. Fish, birds, prey items, eagle bloods are being analyzed for organochlorine pesticides, PCB congeners, non-*ortho*-PCBs, and PCDDs/PCDFs. This report includes the organochlorine and PCB congener results for a selected set of 19 eagle bloods.

The OC-pesticide and 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 PCBs were very low and use of the bovine serum allowed for accurate assessment of the method through matrix spikes. Furthermore, each sample is spiked with appropriate chemical compounds that enable the quality of each analysis to be monitored.

The concentrations of total-PCBs in the eagle serums and bloods ranged from 214 - 14,240 ng/g. Percent lipids ranged from 0.16-1.22%, however there appears to be no correlation between the percent lipid and total-PCB concentration. At this time, the site information remains undisclosed, so correlation to environmental conditions and spatial distributions cannot be made. Patterns of the PCBs in the blood and serum show low amounts of mono-chloro through tri-chloro PCBs and greater amounts of the higher chlorinated congeners. Congeners with the highest concentrations in any of the samples were 153, 138, 118, and 180. Dioxin-like, mono-*ortho* congener concentrations in serum and blood ranged from: 2.8 – 260 ng/g for 105, 0.28 – 29 ng/g for 114, 6.4 – 892 ng/g for 118, 0.1 – 20 ng/g for 123, 0.4 – 122 ng/g for 156, 0.2 – 19 ng/g for 157, and 0.15 – 10 ng/g for 189. Once the non-*ortho* PCB results and the PCDD/PCDF information are available, the total dioxin-like concentrations can be assessed.

Concentrations of p,p'-DDE in serums and bloods ranged from 11 to 986 ng/g. Generally, the only pesticides found in these samples were DDT and chlordane components. Other pesticides—dieldrin and mirex—were found at low levels only. There was no correlation between higher percent lipid levels and higher p,p'-DDE concentrations. Higher DDE concentrations correlated with higher total PCBs.

Acknowledgements

We thank all the chemists of the Organic Chemistry Section whose expertise in conducting trace organic analysis was integral to this research: Kevin Feltz, George

Tegerdine, Mike Tanner, Tim McTague. Their dedication to detailed, high quality, research and analysis is greatly appreciated.

REFERENCES

- 1. CERC SOP P.461. 1998. "Extraction of Animal Tissues for Residue Analysis and Percent Lipid Determination."
- 2. CERC SOP P.196. 1998. "Preparation and Use of Gravity-flow Gel Permeation Chromatography (GPC) Columns."
- 3. CERC SOP P.464. 1998. "Use of High Performance Gel Permeation Chromatography in Sample Preparatory Applications."
- 4. CERC SOP: P.460. 1998. "Organochlorine Pesticide Analysis: Fractionation of Complex Mixtures on Silica Gel/ODS."
- 5. CERC SOP: P.480. 1998. "Automated Porous Graphitic Carbon Column HPLC System for Fractionation of Polychlorinated Biphenyls and Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans."
- 6. CERC SOP: P.195. 1998. "Capillary Gas Chromatography with Electron Capture Detection Procedure for Congener Specific Polychlorinated Biphenyl Analysis."
- Keith, L. H.; Crummet, W; Deegan, J., Jr; Libby, R. A.; Taylor, J. K.; and Wentler, G. 1983. "Principles of environmental analysis." <u>Anal. Chem.</u> 55: 2210-2218.
- 8. Keith, L. H. 1991. Environmental Sampling and Analysis. Lewis Publishers.
- 9. CERC SOP P.483. 1998. "Quality Control Guidelines and Criteria for Gas Chromatographic Data Processing."
- 10. CERC SOP: P.459. 1998. "Organochlorine Pesticide Analysis by High Resolution Capillary Gas Chromatography with Electron Capture Detection."

Chemical Contamination of Nesting Tree Swallows, Great Blue Herons, and Resident/Nesting Bald Eagles Along the Hudson River, New York Report #1: July 20, 2000

Prepared By:

John C. Meadows **Research Chemist**

. .

Prepared By:

Kathy R. Echols Research Chemist

Prepared By:

Und

Paul H. Peterman Chemist, GC/HRMS

Prepared By:

Robert W. Gale Research Chemist

Reviewed By:

Carl E. Orazio, Leader, Organic Chemistry Section USGS Project Officer

Approved By:

Jim D. Petty

Chief, Environmental Chemistry Branch

Approved By:

Paul Heine CERC Quality Assurance Officer

Approved by:

Bill Mauck Director, Columbia Environmental Research Center

Table 4. Organochlorine Pesticides in Eagle Blood (ng/g)

Sample	Field	Species	Total geq	Percent	A-BHC	B-BHC	HCB	PCA	Lindane	D-BHC
Name	ID		for	Lipid						
			Analysis			1				
				· · · · · · · · · · · · · · · · · · ·						
19848-s	BE-A-BL904-98	Eagle Blood	2.65	0.45 +/- 0.08	< 0.01	< 0.4	< 0.8	0.1	< 0.02	< 0.05
19849-s	BE-A-BL949-98	Eagle Blood	3.87	0.56 +/- 0.06	0.1	< 0.4	< 0.8	0.1	< 0.02	< 0.05
19850-s	BE-IM-BL935-98	Eagle Blood	3.25	0.37 +/- 0.07	0.1	< 0.4	1.1	0.4	< 0.02	< 0.05
19851-s	BE-A-BL913-98	Eagle Blood	2.63	0.46 +/- 0.08	< 0.01	< 0.4	0.8	0.3	< 0.02	< 0.05
19852-s	BE-A-BL956-98	Eagle Blood	2.18	1.0 +/- 0.1	< 0.01	< 0.4	1.0	0.2	< 0.02	< 0.05
19853-s	BE-IM-BL914-98	Eagle Blood	0.99	1.22 +/- 0.22	< 0.01	0.6	1.6	0.5	< 0.02	< 0.05
19854-s	BE-BL921-98	Eagle Blood	2.92	0.75 +/- 0.08	< 0.01	0.4	2.0	0.2	< 0.02	< 0.05
19855-s	BE-A-BL898-97	Eagle Blood	3.14	0.38 +/- 0.07	< 0.01	< 0.4	< 0.8	0.1	0.1	< 0.05
19856-s	BE-A-BL968-99	Eagle Blood	1.52	1.11 +/- 0.15	< 0.01	0.7	3.2	0.5	0.1	< 0.05
19857-s	BE-NE-BL950-98	Eagle Blood	3.93	0.93 +/- 0.06	0.1	< 0.4	< 0.8	0.1	< 0.02	< 0.05
19858-s	BE-NE-BL911A-98	Eagle Blood	3.27	0.67 +/- 0.07	0.1	< 0.4	< 0.8	0.2	< 0.02	< 0.05
19859-s	BE-NE-BL911B-98	Eagle Blood	3.43	0.49 +/- 0.06	< 0.01	< 0.4	< 0.8	0.1	< 0.02	< 0.05
19860-s	BE-NE-BL911C-98	Eagle Blood	3.17	0.22 +/- 0.07	< 0.01	< 0.4	< 0.8	0.1	< 0.02	< 0.05
20026-s	BE-IM-BL976-99	Eagle Blood	2.87	0.42 +/- 0.08	0.1	< 0.4	0.9	0.2	0.1	< 0.05
20027-w	BE-NE-BL972A-99	Eagle Blood	3.05	0.55 +/- 0.07	< 0.01	< 0.4	< 0.8	0.1	< 0.02	< 0.05
20028-w	BE-NE-BL972B-99	Eagle Blood	3.16	0.38 +/- 0.07	< 0.01	< 0.4	< 0.8	0.1	< 0.02	< 0.05
20029-w	BE-NE-BL971-99	Eagle Blood	3.08	0.23 +/- 0.07	< 0.01	< 0.4	< 0.8	0.1	< 0.02	< 0.05
20030-w	BE-NE-BL974-99	Eagle Blood	3.05	0.23 +/- 0.07	< 0.01	< 0.4	< 0.8	0.1	< 0.02	< 0.05
20031-w	BE-IM-BL981-99	Eagle Blood	3.50	0.34 +/- 0.06	0.1	< 0.4	0.9	0.1	0.1	< 0.05
s=Blood Serum				· · · · · · · · · ·					· · · · · · · · · · · · · · · · · · ·	• • • • • • •
w=Whole Blood				· · · · · · · · · · · · · · ·	t.					
mass corrected	MDL	average mass =	2.86	grams	0.01	0.4	0.8	0.05	0.02	0.05

1

7/20/00

Table 4. Organochlorine Pesticides in Eagle Blood (ng/g)

Sample	Heptachlor	Dacthal	Heptachlor	Oxychlordane	trans-	o,p-DDE	Endosulfan 1	cis-	trans-	Dieldrin	p,p'-DDE	
Name			Epoxide		Chlordane			Chlordane	Nonachlor			
19848-s	< 0.01	< 3.3	02	1.0	< 0.3	< 0.01	< 0.01	04	34	15	79	
19849-s	< 0.01	< 3.3	0.5	1.6	< 0.3	< 0.01	< 0.01	0.4	5.0	2.0	110	
19850-s	< 0.01	< 3.3	2.5	4.6	0.7	0.1	< 0.01	2.7	16	9.0	210	
19851-s	< 0.01	< 3.3	4.3	6.8	0.4	0.1	< 0.01	1.2	17	16	260	
19852-s	< 0.01	< 3.3	0.6	2.0	< 0.3	< 0.01	< 0.01	0.7	6.9	2.5	150	
19853-s	< 0.01	< 3.3	0.7	2.2	0.6	0.1	< 0.01	4.6	12	3.9	160	
19854-s	0.1	< 3.3	1.3	1.5	0.4	0.1	< 0.01	3.1	10	9.1	99	
19855-s	< 0.01	< 3.3	1.5	4.7	< 0.3	< 0.01	< 0.01	1.2	21	9.5	350	1.1.18
19856-s	0.8	< 3.3	3.8	11	1.1	0.5	0.5	6.4	59	28	990	KI LA
19857-s	0.1	< 3.3	0.1	0.4	0.3	< 0.01	< 0.01	0.9	3.1	1.4	64	, ·
19858-s	< 0.01	< 3.3	0.1	0.3	0.3	0.1	< 0.01	1.0	1.8	1.1	21	
19859-s	< 0.01	< 3.3	0.1	0.2	< 0.3	< 0.01	< 0.01	0.6	1.3	0.7	14	
19860-s	< 0.01	< 3.3	0.1	0.1	< 0.3	< 0.01	< 0.01	0.6	1.0	0.7	11	
20026-s	< 0.01	< 3.3	2.9	4.3	1.3	0.2	0.2	7.7	21	24	870	may in as
20027-w	< 0.01	< 3.3	0.1	0.3	0.3	0.1	< 0.01	0.8	1.6	1.0	27	· C
20028-w	< 0.01	< 3.3	0.1	0.2	< 0.3	< 0.01	< 0.01	0.7	1.5	0.9	22	
20029-w	< 0.01	< 3.3	0.1	0.1	< 0.3	< 0.01	< 0.01	0.5	0.8	0.7	16	
20030-w	< 0.01	< 3.3	0.1	0.2	< 0.3	< 0.01	< 0.01	0.5	1.3	0.8	20	
20031-w	< 0.01	< 3.3	0.7	1.7	0.3	0.1	< 0.01	1.7	4.8	3.8	91	
s=Blood Serum							· · · · •					
w=Whole Blood				-								
mass corrected M	0.01	3.3	0.03	0.1	0.3	0.01	0.01	0.01	0.01	0.2	0.5	

7/20/00

Sample Name	o,p'-DDD	Endrin	Endosulfan 2	p,p'-DDD	<i>cis-</i> Nonachlor	o,p'-DDT	Endosulfate	p,p'-DDT	Methoxychlor	Mirex
10040									· · · · · · · · · · · · · · · · · · ·	
19848-5	0.5	0.3	< 0.01	1.2	1.0	< 0.01	< 0.5	0.9	< 1.5	1.7
19849-5	0.4	0.1	0.1	1.9	1.5	0.4	< 0.5	0.6	< 1.5	3.4
19850-s	0.2	0.5	0.1	6.2	5.9	1.1	< 0.5	0.7	< 1.5	12
19851-s	0.1	0.5	0.3	2.2	3.6	1.5	< 0.5	0.9	< 1.5	25
19852-s	0.1	< 0.1	< 0.01	2.6	2.0	0.8	< 0.5	1.0	< 1.5	2.5
19853-s	0.3	0.1	0.1	11	4.5	0.8	< 0.5	6.9	< 1.5	1.1
19854-s	0.3	0.1	0.1	7.1	4.4	0.9	< 0.5	0.8	< 1.5	2.1
19855-s	0.1	0.5	0.2	9.5	6.3	0.4	< 0.5	2.1	< 1.5	8.3
19856-s	0.8	< 0.1	1.0	47	19	2.3	1.0	4.3	< 1.5	7.3
19857-s	0.3	< 0.1	< 0.01	· 3.8	0.9	0.1	< 0.5	0.7	< 1.5	0.3
19858-s	0.2	< 0.1	0.1	2.2	0.7	0.1	< 0.5	0.7	< 1.5	0.2
19859-s	0.0	< 0.1	< 0.01	1.3	0.5	< 0.01	< 0.5	0.6	< 1.5	0.2
19860-s	0.2	< 0.1	< 0.01	1.2	0.4	< 0.01	< 0.5	0.8	< 1.5	0.1
20026-s	1.9	0.8	2.2	80	12	1.9	< 0.5	2.8	< 1.5	3.5
20027-w	0.4	< 0.1	< 0.01	2.3	0.6	0.1	< 0.5	0.7	< 1.5	0.2
20028-w	0.3	< 0.1	< 0.01	1.7	0.4	< 0.01	< 0.5	0.6	< 1.5	0.2
20029-w	0.1	< 0.1	< 0.01	1.2	0.3	< 0.01	< 0.5	< 0.5	< 1.5	< 0.1
20030-w	0.3	0.1	< 0.01	1.5	0.5	< 0.01	< 0.5	0.9	< 1.5	0.3
20031-w	0.7	0.6	0.2	6.0	1.9	0.2	< 0.5	0.9	< 1.5	2.0
s=Blood Serum							· · · · ·			
w=Whole Blood										
mass corrected N	0.6	0.1	0.01	0.01	0.05	0.01	0.5	0.5	1.5	0.1

Table 4. Organochlorine Pesticides in Eagle Blood (ng/g)

USDI, USGS, BRD, CERC, FinalEBOCs.xls, Table 4-Final OC sample

7/20/00

Table 5. Organochlorine Pesticide QC--Eagle Blood (ng)

all in ng amounts			I I		1					
Sample	Field	Species	Gram equivalents	Percent	A-BHC	B-BHC	HCB	PCA	Lindane	D-BHC
Name	ID		for	Lipid						
			Analysis	· •						
			• • •							
MS102699#1 GCR1 (ng)	Matrix Spike	Bovine Serum	2.87	< 0.15	22	28	19	28	24	25
MS102699#1 GCR2 (ng)	Matrix Spike	Bovine Serum	2.87	< 0.15	25	32	19	31	28	28
MS102699#1 GCR3 (ng)	Matrix Spike	Bovine Serum	2.87	< 0.15	26	33	18	31	28	29
Average					26	33	18	31	28	29
SD (n-1)				1	0.3	0.4	0.0	0.2	0.3	0.2
% RSD					1.3	1.4	0.1	0.7	1.2	0.7
Percent Recovery	· · · · · · · · · · · · · · · · · · ·			- -	75	82	41	68	80	90
MB102699#1 (ng/g)	Matrix Blank	Bovine Serum	2.94	< 0.15	< 0.01	< 0.4	< 0.8	< 0.05	< 0.02	< 0.05
MB102699#2 (ng/g)	Matrix Blank	Bovine Serum	2.94	< 0.15	< 0.01	< 0.4	< 0.8	< 0.05	< 0.02	< 0.05
PB102699 GCR1 (ng)	Procedural Blank	Na₂SO₄	· · · · · · · · ·		0.00	0.20	1.05	0.04	0.03	0.06
PB102699 GCR2 (ng)	Procedural Blank	Na ₂ SO ₄			0.00	0.47	1.05	0.02	0.03	0.07
PB102699 GCR3 (ng)	Procedural Blank	Na₂SO₄	••••		0.00	0.48	1.08	0.02	0.05	0.08
PB102999 GCR1 (ng)	Procedural Blank	Na ₂ SO ₄			0.00	0.63	1.61	0.11	0.03	0.00
PB102999 GCR2 (ng)	Procedural Blank	Na ₂ SO ₄			0.00	0.77	1.61	0.03	0.03	0.00
PB102999 GCR3 (ng)	Procedural Blank	Na ₂ SO ₄		1	0.00	0.80	1.62	0.03	0.03	0.00
Average				*	0.00	0.56	1.34	0.04	0.03	0.03
SD (n-1)			•		0.00	0.22	0.30	0.03	0.01	0.04
MDL=PB AVG+3(PB SD)	Method Detection Limit		1	• • • •	0.00	1.23	2.25	0.14	0.06	0.15
mass normalized MDL		average mass =	2.86	grams	0.01	0.4	0.8	0.05	0.02	0.05

305045

USDI, USGS, BRD, CERC, FinalEBOCs.xls, Table 5 QAQC

7/20/00

Table 5. Organochlorine Pesticide QC--Eagle Blood (ng)

. all in ng amounts				1	1	- 1		1			
Sample	Heptachlor	Dacthal	Heptachlor	Oxychiordane	trans-	o,p-DDE	Endosulfan 1	cis-	trans-	Dieldrin	p,p'-DDE
Name		· · · · · · · · · · · · · · · · · · ·	Epoxide		Chlordane		ан алан алан алан алан алан алан алан а	Chlordane	Nonachlor		·····
											· · · · · · · · · · · · · · · · · · ·
MS102099#1 GCR1 (ng)	11	29	27	28	27	28	20	28	29	20	20
MS102699#1 GCH2 (ng)	12	36	33	34	32	31	31	32	34	32	20
MS102699#1 GCH3 (ng)	12		34	35	33	22	31	33	35	33	20
Average	12	36	34	35	32	32	31	33	35	32	20
SD (n-1)	0.2	0.5	0.7	0.8	0.6	0.5	0.6	0.6	0.7	0.6	0.4
% RSD	1.7	1.5	2.2	2.3	1.8	1.6	2.1	1.8	2.0	1.8	2.0
Percent Recovery	26	84	84	80	84	86	82	83	86	85	61
MB102699#1 (ng/g)	< 0.01	< 3.3	< 0.03	< 0.1	< 0.3	0.01	0.02	0.01	< 0.01	< 0.2	< 0.5
MB102699#2 (ng/g)	0.2	< 3.3	< 0.03	< 0.1	< 0.3	< 0.01	0.02	< 0.01	< 0.01	< 0.2	< 0.5
PB102699 GCR1 (ng)	0.00	2.20	0.00	0.13	0.03	0.00	0.00	0.01	0.02	0.01	0.97
PB102699 GCR2 (ng)	0.02	2.34	0.00	0.02	0.01	0.01	0.00	0.01	0.02	0.16	0.98
PB102699 GCR3 (ng)	0.03	2.25	0.05	0.02	0.02	0.00	0.00	0.03	0.02	0.13	1.08
PB102999 GCR1 (ng)	0.00	5.28	0.00	0.25	0.06	0.02	0.00	0.02	0.01	0.22	0.56
PB102999 GCR2 (ng)	0.00	5.49	0.00	0.00	0.06	0.00	0.01	0.03	0.01	0.27	0.58
PB102999 GCR3 (ng)	0.00	5.84	0.00	0.00	0.58	0.00	0.00	0.02	0.02	0.33	0.58
Average	0.01	3.90	0.01	0.07	0.13	0.01	0.00	0.02	0.02	0.18	0.79
SD (n-1)	0.01	1.80	0.02	0.10	0.22	0.01	0.00	0.01	0.00	0.11	0.24
MDL=PB AVG+3(PB SD)	0.04	9.30	0.07	0.37	0.80	0.03	0.01	0.04	0.03	0.51	1.52
mass normalized MDL	0.01	3.3	0.03	0.1	0.3	0.01	0.01	0.01	0.01	0.2	0.5

7/20/00

Table 5. Organochlorine Pesticide QC--Eagle Blood (ng)

all in ng amounts					,				•	
Sample	o,p'-DDD	Endrin	Endosulfan 2	p,p'-DDD	cis-	o,p'-DDT	Endosulfate	p,p'-DDT	Methoxychior	Mirex
Name				· · · · · ·	Nonachlor	·				
MS102600#1 GCB1 (ng)		10.3		· · · · · · · · · · · · · · · · · · · ·					28	
MS102039#1 GORT (ng)	21	20.0		 30		6 7 j 35 j	20	20	38	24
MS102055#1 GON2 (ng)	34	23.7	34	31	34	37	34	37	43	25
NOTO2033#1 GOILO (19)	34	23.2	33	30	34	36	33	35		25
Prelage	05	07	0.7	0.4	07	16	1.0	21	32	0.5
% RSD	1.5	28		14	20	4.4	3.0	61	80	21
Percent Recovery	84	58	. 85	95	87	93	87	105	113	45
MB102699#1 (ng/g)	< 0.6	< 0.1	< 0.01	< 0.01	< 0.05	< 0.01	< 0.5	0.7	< 1.5	< 0.1
MB102699#2 (ng/g)	< 0.6	< 0.1	< 0.01	< 0.01	< 0.05	< 0.01	< 0.5	0.6	< 1.5	< 0.1
PB102699 GCR1 (ng)	0.24	0.01	0.00	0.00	0.04	0.00	0.12	0.63	0.10	0.11
PB102699 GCR2 (ng)	0.25	0.03	0.00	0.00	0.05	0.00	0.14	0.69	0.06	0.11
PB102699 GCR3 (ng)	0.25	0.00	0.00	0.02	0.03	0.00	0.13	0.76	0.18	0.11
PB102999 GCR1 (ng)	0.95	0.26	0.02	0.01	0.08	0.01	0.89	1.12	2.23	0.04
PB102999 GCR2 (ng)	0.95	0.08	0.00	0.01	0.09	0.02	0.74	1.03	2.22	0.06
PB102999 GCR3 (ng)	0.90	0.03	0.00	0.00	0.02	0.00	0.72	1.08	1.76	0.05
Average	0.59	0.07	0.00	0.01	0.05	0.01	0.45	0.88	1.09	0.08
SD (n-1)	0.38	0.10	0.01	0.01	0.03	0.01	0.36	0.22	1.08	0.04
MDL=PB AVG+3(PB SD)	1.72	0.36	0.02	0.03	0.13	0.03	1.54	1.53	4.35	0.19
mass normalized MDL	0.6	0.1	0.01	0.01	0.05	0.01	0.5	0.5	1.5	0.1
	•••	· · · · · · · · · · · · · · · · · · ·		. .						

305047

7/20/00

Table 6. Procedural Recoveries for OC Pesticides

Sample	Field	Species	TOTAL geq	PCB 029	%	PCB 204	%	DBC	%
Name	ID		for		Recovery	•	Recovery		Recovery
	· · · · · · · · · · · · · · · · · · ·	•	Analysis		••••••				•
	1							بسنو ويت المنصاب	-
19848-s	BE-A-BL904-98	Eagle Blood	2.65	6.0	38	11	71	16	98
19849-s	BE-A-BL949-98	Eagle Blood	3.87	3.8	35	7.6	73	9.2	85
19850-s	BE-IM-BL935-98	Eagle Blood	3.25	6.5	50	8.9	72	11	85
19851-s	BE-A-BL913-98	Eagle Blood	2.63	8.6	54	11	71	12	74
19852-s	BE-A-BL956-98	Eagle Blood	2.18	9.2	48	12	63	17	89
19853-s	BE-IM-BL914-98	Eagle Blood	0.99	13	31	27	67	41	97
19854-s	BE-BL921-98	Eagle Blood	2.92	6.5	45	10	76	13	89
19855-s	BE-A-BL898-97	Eagle Blood	3.14	6.1	45	9.0	70	11	82
19856-s	BE-A-BL968-99	Eagle Blood	1.52	12	43	18	67	20	73
19857-8	BE-NE-BL950-98	Eagle Blood	3.93	3.6	34	7.1	69	9.7	91
19858-s	BE-NE-BL911A-98	Eagle Blood	3.27	7.2	56	9.2	75	12	94
19859-s	BE-NE-BL911B-98	Eagle Blood	3.43	6.2	50	8.1	69	12	98
19860-s	BE-NE-BL911C-98	Eagle Blood	3.17	7.3	55	9.1	72	13	98
20026-s	BE-IM-BL976-99	Eagle Blood	2.87	5.5	38	10	72	12	83
20027-w	BE-NE-BL972A-99	Eagle Blood	3.05	5.4	39	9.5	72	14	100
20028-w	BE-NE-BL972B-99	Eagle Blood	3.16	5.0	38	9.2	72	13	97
20029-w	BE-NE-BL971-99	Eagle Blood	3.08	7.6	56	10	7A	13	98
20030-w	BE-NE-BL974-99	Eagle Blood	3.05	3.9	29	10	75	13	9 3
20031-w	BE-IM-BL981-99	Eagle Blood	3.50	4.0	33	2.3	72	11	88
MB102699#1	Matrix Blank	Bovine Serum	2.94	5.7	40	9.9	72	39	92
MB102699#2	Matrix Blank	Bovine Serum	2.94	4.2	30	10	75	39	93
MS102699#1	Matrix Spike	Bovine Serum	2.96	18	43	26	65	40	95
PB102699	Procedural Blank	Na ₂ SO ₄	•••	12	29	27	66	31	73
PB102999	Procedural Blank	Na ₂ SO ₄	•-•	18	44	29	71	35	85
Average		1			42		71		90
Std Dev.	• • •		•		: 9 [:]		4		8

7/20/00