Rec 9/3/17

2,3,7,8-Tetrachlorodibenzo-p-dioxin Concentrations in Double-crested Cormorant and Black-crowned Night-Heron Eggs of Shooters Island and Isle of Meadows, New York.

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Introduction

Shooter's Island has the distinction of being the cornerstone of wading bird and other breeding waterbird populations in the New York Bight/Arthur Kill ecosystem, and has been designated as a coastal habitat of unique significance (U.S. Dept. Commerce 1982). Productive shallow-water habitats in close proximity to other suitable nesting islands in the area explain the island's significance to the ecosystem's waterbird populations. It is estimated that heron populations in the New York Harbor represent about 25% of all nesting waders along the coast from Cape May, New Jersey, to Rhode Island (Parsons 1994). The Isle of Meadows has the distinction of being designated as a priority wetland site by U.S. Fish and Wildlife Service (Service).

Another distinction for Shooter's Island and the Isle of Meadows is that they are located in one of the most degraded estuaries in North America (Squibb *et al.* 1991). More than 40 contaminants have been identified in the water, sediments, and biota of the New York Bight/Arthur Kill ecosystem that exceed federal and state standards for protecting human and wildlife health. Of particular concern are the exceedingly high levels of 2,3,7,8 tetrachlorodibenzo-p-dioxin (TCDD), which have been found in the sediments, fish, and shellfish of the area (NYSDEC 1987, Rappe *et al.* 1991, Squibb 1991, and Wellman 1993).

Dioxins are included in a group of compounds collectively known as planar chlorinated hydrocarbons (PCHs). These compounds are persistent, synthetic, chlorinated hydrocarbons which are known for their potential for bioaccumulation, and appear to be toxic at low concentrations. It is believed that their toxicity is expressed through binding of the PCH molecule to the arylhydrocarbon receptor (AhR), a ligand activated transcription factor found in most vertebrates (Safe 1987, Goldstein 1980, and McFarland and Clarke 1989). Among the PCHs, TCDD has the greatest affinity for the AhR and is considered to be the most toxic of the PCHs (Hoffman *et al.* 1995, Ahlborg *et al.* 1992). Toxic effects in birds and mammals include decreased reproductive success, immunotoxicity, hepatotoxicity, enzyme induction, altered biochemical homeostasis, embryotoxicity, teratogenesis, promotion of cancer and chick edema disease (Kimbrough 1974, Poland et al 1979, Poland and Knutson 1982, Gilbertson *et al.* 1991, Kociba and Schwetz 1982, Safe 1984, Nikolaidis *et al.* 1988, and Nosek *et al.* 1992).

The implications for exposure of the waterbirds in the New York Bight ecosystem to the observed levels of PCHs in sediment, shellfish, and fish are unclear, but comparable concentrations in Great Lakes sediments have been associated with reduced reproductive success (USEPA 1993) and other symptoms of PCH intoxication (Gilbertson 1983, Gilbertson *et al.* 1991, and Brunstrom 1988) in piscivorous birds.

The Service's Hudson River/New York Bight Ecosystem Team has established as a high priority, that the Service "compile existing information, conduct injury assessments and develop recommendations to reduce concentrations of dioxins and other contaminants in the Passaic River, Newark Bay and Arthur Kill, especially contamination from the Diamond Alkali Superfund Site to restore public use of the area and ensure viable populations of fish and wildlife".

Towards that end, this study was conducted to determine:

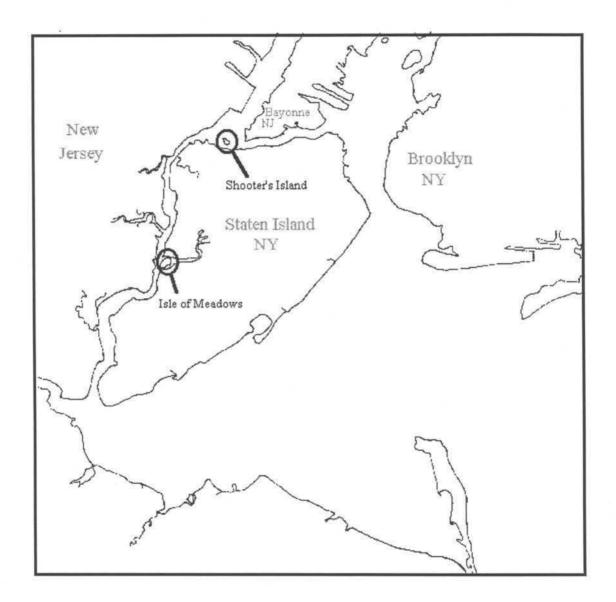
- 1. dioxin concentrations in samples of Double-crested Cormorant (*Phalacrocorax auritus*) and Black-crowned Night-Heron (*Nycticorax nycticorax*) eggs collected in the New York Bight ecosystem, and
- assess the potential risk to fish-eating birds of the ecosystem from exposure to dioxin and other PCHs.

Study Area and Methods

Shooter's Island (40° 38' 35" N, 74° 9' 36" W, Richmond County, New York, Hudson and Union Counties, New Jersey) is located in the New York Bight at the confluence of Newark Bay, Kill Van Kull, and Arthur Kill. Isle of Meadows (40° 34' 32" N, 74° 12' 15" W, Richmond County, New York) is located along the Arthur Kill in Staten Island, New York, east of Carteret, New Jersey (Figure 1).

Double-crested Cormorant and Black-crowned Night-Heron eggs were collected in 1993 (and archived) from randomly selected nests located at Shooter's Island and the Isle of Meadows, respectively. Eggs were refrigerated upon field collection and the contents harvested within 48 hr. Egg contents were stored frozen (-86 C°) in chemically-clean glass jars until submitted for a full dioxin scan. An analyte list is given in Table 1. Laboratory results of the sample analyses are reported in Appendix A.

Residue analyses were conducted by the Geochemical and Environmental Research Group, Texas A&M University, College Station, TX. Quality assurance/quality control (QA/QC) for the analytical techniques used by the contract laboratory were established and overseen by the Service's Patuxent Analytical Control Facility (PACF). Analytical methodologies are provided Appendix B.



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Figure 1. Location of Shooter's Island and the Isle of Meadows within the New York Bight study area.

Table 1. Compounds included in the "Full Dioxin Scan" as specified by the Patuxent Analytical Control Facility.

IUPAC ¹	Based	Nomenc	lature
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Abbreviated Name

2,3,7,8- Tetrachlorodibenzo	2,3,7,8- TCDD
2,3,7,8- Tetrachlorodibenzo-p-Dioxin	2,3,7,8- TCDF
1,2,3,7,8- Pentachlorodibenzo-p-Dioxin	1,2,3,7,8- PeCDD
1,2,3,4,7,8- Hexachlorodibenzo-p-Dioxin	1,2,3,4,7,8- HxCDD
1,2,3,6,7,8- Hexachlorodibenzo-p-Dioxin	1,2,3,6,7,8- HxCDD
1,2,3,7,8,9- Hexachlorodibenzo-p-Dioxin	1,2,3,7,8,9- HxCDD
1,2,3,4,6,7,8- Heptachlorodibenzo-p-Dioxin	1,2,3,4,6,7,8- HpCDD
Octachlorodibenzo-p-Dioxin	OCDD
1,2,3,7,8- Pentachlorodibenzofuran	1,2,3,7,8- PeCDF
2,3,4,7,8- Pentachlorodibenzofuran	2,3,4,7,8- PeCDF
1,2,3,4,7,8- Hexachlorodibenzofuran	1,2,3,4,7,8- HxCDF
1,2,3,6,7,8- Hexachlorodibenzofuran	1,2,3,6,7,8- HxCDF
1,2,3,7,8,9- Hexachlorodibenzofuran	1,2,3,7,8,9- HxCDF
2,3,4,6,7,8- Hexachlorodibenzofuran	2,3,4,6,7,8- HxCDF
1,2,3,4,6,7,8- Heptachlorodibenzofuran	1,2,3,4,6,7,8- HpCDF
1,2,3,4,7,8,9- Heptachlorodibenzofuran	1,2,3,4,7,8,9- HpCDF
Octachlorodibenzofuran	OCDF

¹ IUPAC - International Union of Pure and Applied Chemistry ; naming rules as applied in <u>Nomenclature of Organic Chemistry</u>, 1979 Edition, published by Pergamon Press, Inc.

Contaminant concentrations are reported in parts per trillion (pg/g) wet weight unless otherwise noted. Residues for each sample are reported when at least half of the samples had detectable levels of contaminants. For this report, a value of one-half the detection limit was assigned as the sample concentration for results below the detection limit. Residue concentrations were log transformed prior to statistical analysis and the re-transformed means are presented as the geometric mean in the tables.

Results and Discussion

A total of eight (8) Double-crested Cormorant and 10 Black-crowned Night-Heron eggs from Shooter's Island and the Isle of Meadows, respectively, were submitted for residue analysis. Among the dioxins and furans included in the dioxin scan (Table 1), 2,3,7,8 TCDD was the only analyte that occurred in more than half of the samples analyzed.

2,3,7,8 TCDD occurred in detectable levels in every cormorant egg collected from Shooter's Island (min. = 46.5 pg/g; max. =161 pg/g), and in eight of the ten heron eggs collected from the Isle of Meadows (min. = non-detected; max. =86.8 pg/g). A summary of those results is given in Table 2. Concentrations of all other compounds included in the Dioxin Scan (Table 1) were below the level of detection, with the exception of OCDD. OCDD was detected in two (2) of the 10 Black-crowned Night-Heron egg samples analyzed (Shot9513, 73.2 pg/g, LOD=65.36 pg/g; Shot9514, 85.1 pg/g, LOD=65.79 pg/g). Because OCDD was detected in less than 50 percent of the samples, it was excluded from further consideration.

In order to assess the potential for adverse effects in fish-eating birds in the New York Bight due to exposure to TCDD, a comparison of several toxicity threshold concentrations can be made to the levels found in the cormorant and heron eggs collected (After Frakes and Lemieux In Prep). These include the: (1) lowest observed adverse effect level (LOAEL) in sensitive avian species; (2) documented effects in populations of wild birds; and, (3) 50 percent embryonic mortality (or LD₅₀ - "Median Lethal Dose; The quantity of a chemical compound which, when applied directly to test organisms, is estimated to be fatal to 50 percent of those organisms under the stated conditions of the test". (Hodgson and Levi. 1987. Modern Toxicology. Elsevier Sci. Pub. Co., Inc. NY, NY.).

In this report a value of 10 pg/g TCDD is used as the LOAEL. It is a value which has been shown to cause embryonic mortality, edema, and teratogenic effects in the most sensitive avian species, i.e. the chicken (Verrett 1970). It is also the value adopted in the Great Lakes Initiative to evaluate the sublethal effects of TCDD (USEPA 1995). Measured values above 10 pg/g would be predictive of adverse effects in sensitive avian species; 50 pg/g TCDD in eggs is used as the toxicity threshold value for documented effects in wild birds since this level in the eggs of Great Blue Herons, Wood Duck, Forster's Tern, and Double-crested Cormorant and has been associated with effects on reproduction or development (Elliott *et al.* 1989; Tillitt *et al.* 1992, 1993; and White and Seginak 1994) and finally, the LD_{50} value of 147 pg/g TCDD is used as the third toxicity threshold value, since it represents 50 percent embryonic mortality and is the basis for calculating Toxic Equivalency Factors (Verrett 1976, Hoffman *et al.* 1996).

Comparisons of the TCDD concentrations in the eggs from both species to the three aforementioned reference toxicity values (10 pg/g, 50 pg/g and 147 pg/g) are summarized in Figure 1, Figure 2, and Figure, 3 respectively. The concentration of TCDD in every cormorant egg (100%) from Shooter's Island exceeded the LOAEL (10 pg/g), and on average, was 7.5 times greater than that threshold value for sensitive avian species (i.e. exceeded levels which would be expected to have adverse effects by 7.5 times). Seventy-five percent (75%) of the cormorant eggs exceeded the documented effects level for wild birds (50 pg/g) by 1.5 times, and approximately nine percent (9%) had levels 0.5 times as great as the LD₅₀ value of 147 pg/g. The concentration of TCDD in heron eggs collected from the Isle of Meadows exceed the LOAEL on average by 1.34 times, was 0.27 times as great as the effects level for wild birds (50 pg/g), and was 0.09 times as great as the LD₅₀ value (147 pg/g).

From the results of this survey and the above comparison, cormorants appear to be more at risk given the higher mean TCDD levels in their eggs. One reason for this difference may be due to the location of the two sample sites relative to dioxin contaminated sites (which would influence the foraging areas used by nesting birds thus affecting relative exposure). Or more likely, the difference is related to the dietary habits of the two species, cormorants being nearly obligate fish feeders and night-herons feeding more broadly and opportunistically in aquatic, semi-aquatic, and terrestrial habitats (Parsons Per. Comm.).

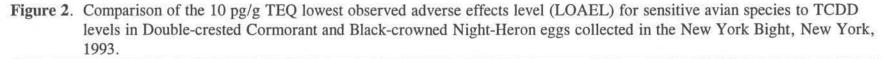
Based on the comparison of TCDD levels in eggs to reference toxicity values determined for other bird species, the measured concentrations of dioxin may be sufficiently high to have adverse impacts on the cormorant and heron populations in the New York Bight. Tillett *et al.* (1992) found increased egg mortality in Double-crested Cormorants beginning at TCDD equivalent (TEQ) concentrations of about 85 pg/g. The geometric mean value of 2,3,7,8-TCDD in cormorant eggs alone for this study was 75.2 pg/g. No data was available to account for the relative toxicity of other dioxin-like compounds (which have a similar binding affinity to the AhR and hence contributory toxicity), specifically the nonortho and mono-ortho-chloro substituted polychlorinated biphenyls (PCBs) (congeners: PCB 77, PCB 105, PCB 118, PCB 126, PCB 156, and PCB 169).

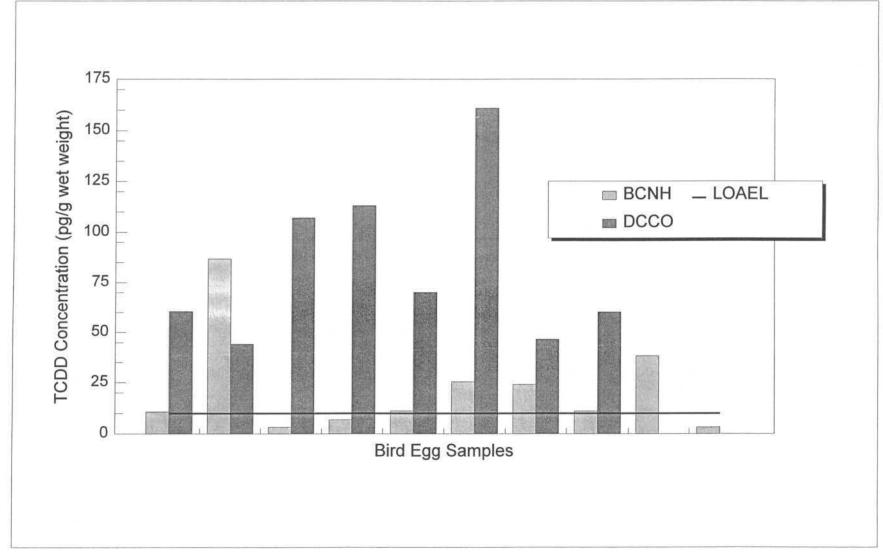
The geometric mean concentration of total PCB in cormorant eggs was 5.72 mg/kg, and in heron eggs was 2.53 mg/kg. Since non-ortho and mono-ortho-chloro substituted PCBs may constitute a large percentage to the total PCBs measured, a greater risk to fish-eating birds in the New York Bight exists than was addressed in this survey. Additional study to investigate possible effects resulting from TCDD and other PCH exposure for the area is warranted. Future studies should incorporate dioxin-like PCBs in the analytes tested for to account for the possibility of additional toxicity based on an evaluation of TEQ.

 Table 2. Dioxin levels in Double-crested Cormorant and Black-crowned Night-Heron eggs collected at Shooter's Island and the Isle of Meadows, New York Bight study area.

Catalog					2,3,7,8-TCD	D
Sample ID	Species	Sample Location	% Lipid	% Moisture	(ppt Wet weight)	Detection Limit
shot9501	DCCO	Shooter's Is	2.44	87.15	60.5	6.5
shot9502	DCCO	Shooter's Is	2.83	86.40	44.1	6.1
shot9503	DCCO	Shooter's Is	2.52	89.66	107	6.5
shot9504	DCCO	Shooter's Is	1.76	86.73	113	6.1
shot9505	DCCO	Shooter's Is	2.36	87.30	70.0	6.4
shot9506	DCCO	Shooter's Is	1.54	88.30	161	6.3
shot9507	DCCO	Shooter's Is	1.72	87.83	46.5	6.6
shot9508	DCCO	Shooter's Is	2.45	86.89	60.2	6.6
			Sample Stan Sa 95 % C	eometric Mean dard Deviation mple Variance .I. Lower Limit .I. Upper Limit	75.15 40.71 1657.17 46.94 103.36	
Catalog					2,3,7,8-TCDI	0
Sample ID	Species	Sample Location	% Lipid	% Moisture	(ppt Wet weight)	Detection Limit
shot9509	BCNH	Isle of Meadows	3.37	86.49	10.7	6.54
shot9510	BCNH	Isle of Meadows	3.76	83.83	86.8	6.5
* shot9511	BCNH	Isle of Meadows	3.85	84.81	3.25	6.4
shot9512	BCNH	Isle of Meadows	2.67	85.47	6.84	6.2
shot9513	BCNH	Isle of Meadows	2.65	84.08	11.1	6.54
shot9514	BCNH	Isle of Meadows	5.40	82.81	25.4	6.5
shot9515	BCNH	Isle of Meadows	3.80	84.85	24.0	6.5
shot9516	BCNH	Isle of Meadows	6.01	85.69	11.0	6.6
shot9517	BCNH	Isle of Meadows	4.07	84.42	38.3	6.6
* shot9518	BCNH	Isle of Meadows	3.59	86.37	3.21	6.4
	8		Sample Stand		13.41 25.35	
				nple Variance .I. Lower Limit	642.51 -4.15	
				I. Upper Limit	30.98	

* Sample concentration below level of detection





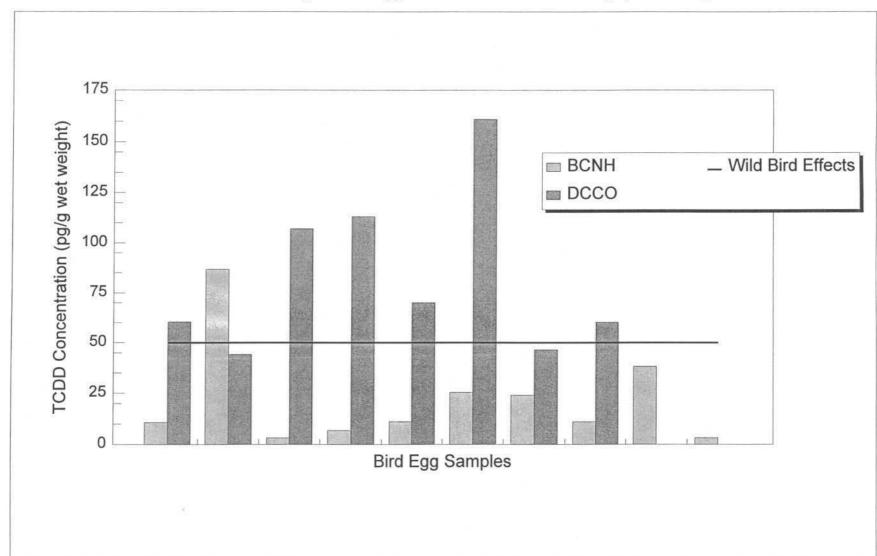


Figure 3. Comparison of the documented 50 pg/g TEQ effect level for wild avian species to TCDD levels in Double-crested Cormorant and Black-crowned Night-Heron eggs collected in the New York Bight, New York, 1993.

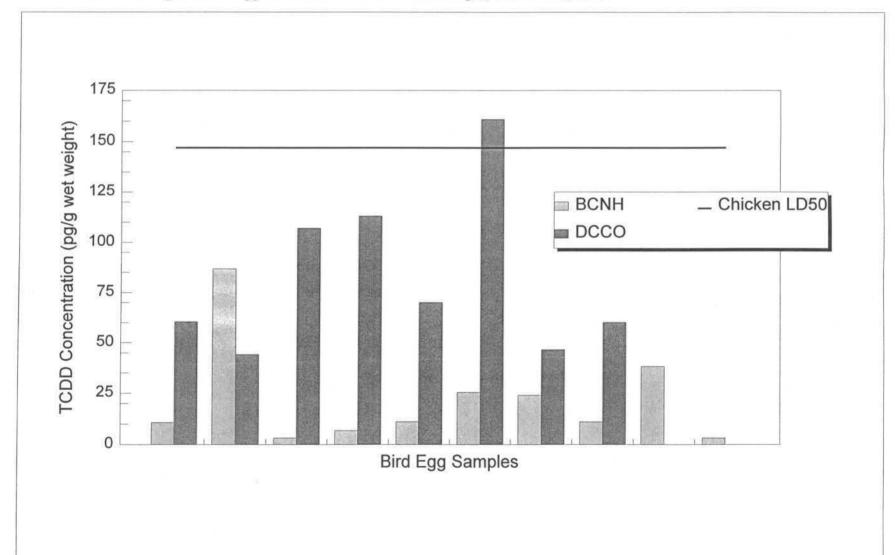


Figure 4. Comparison of the 147 pg/g TEQ effect level for chicken eggs to TCDD levels in Double-crested Cormorant and Blackcrowned Night-Heron eggs collected in the New York Bight, New York, 1993.

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Appendix A. Analytical Results.

ECDMS ANALYTICAL REPORT (6)

1/10/97

Catalog: 5050045 Regional Study Id: 5F19 Purchase Order: 84055-5-3486

User Id: R5CFO Submitter: John Hickey - Cortland, NY

Lab Name: Geochemical & Environmental Research Group, Texas A&M (GERG)

Report Includes the Following Sections:

- Weight, % Moisture, % Lipid, % Ash, Total Suspended Solids
- Soil / Sediment Parameters
- Contaminant Concentrations
- Procedural Blanks
- Duplicates
- Reference Materials
- Spike Recoveries
- Comments (Result Modifers and QA/QC Comments)
- Analytical Methods

Catalog: 5050045 Lab Name: GERG 1/10/97

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WEIGHT, % MOISTURE, % LIPID, % ASH, TOTAL SUSPENDED SOLIDS

Sample Number	Sample Matrix	Weight (g)	Percent Moisture	Percent Lipid	Percent Ash	Total Suspended Solids (%)
shot9501	Avian Egg	87.15	2.44			
shot9502	Avian Egg	86.40	2.83			
shot9503	Avian Egg	89.66	2.52			
shot9504	Avian Egg	86.73	1.76			
shot9505	Avian Egg	87.30	2.36			
shot9506	Avian Egg	88.30	1.54			
shot9507	Avian Egg	87.83	1.72			
shot9508	Avian Egg	86.89	2.45			
shot9509	Avian Egg	86.49	3.37			
shot9510	Avian Egg	83.83	3.76			
shot9511	Avian Egg	84.81	3.85			
shot9513	Avian Egg	84.08	2.65			
shot9514	Avian Egg	82.81	5.40			
shot9515	Avian Egg	84.85	3.80			
shot9516	Avian Egg	85.69	6.01			
shot9517	Avian Egg	84.42	4.07			
shot9518	Avian Egg	86.37	3.59			

Page: 1

SOIL / SEDIMENT PARAMETERS

Sample	Percent	Percent		- Particle Size	
Number	TVS	TOC	%Sand	%Silt	%Clay

- NO DATA EXIST FOR THIS SECTION.

CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,4,6,7,8-HpCDD

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
Number shot9501 shot9502 shot9503 shot9504 shot9505 shot9506 shot9507 shot9508 shot9509 shot9510 shot9511 shot9512	Matrix Avian Egg Avian Egg	<pre>(ppm Dry Weight) </pre> < .00025432 < .00022694 < .00031605 < .00023259 < .00025565 < .0002722 < .00027208 < .00027208 < .00025426 < .00024189 < .00020343 < .00021374 < .00021642	Limit .00025432 .00022694 .00031605 .00023259 .00025565 .0002722 .00027208 .00027208 .00025426 .00024189 .00020343 .00021374 .00021642	<pre>(ppm Wet Weight) </pre> < .00003268 < .00003086 < .00003268 < .00003268 < .00003247 < .00003247 < .00003185 < .00003333 < .00003268 < .00003268 < .00003289 < .00003247 < .00003247 < .00003145	Limit .00003268 .00003086 .00003268 .00003268 .00003247 .00003247 .00003311 .00003333 .00003268 .00003289 .00003247 .00003247 .00003145	4
shot9513 shot9514 shot9515 shot9516 shot9517 shot9518	Avian Egg Avian Egg Avian Egg Avian Egg Avian Egg Avian Egg	< .00020527 < .00019136 < .00021713 < .00023139 < .00021253 < .00023515	.00020527 .00019136 .00021713 .00023139 .00021253 .00023515	<.00003268 <.00003289 <.00003289 <.00003311 <.00003311 <.00003205	.00003268 .00003289 .00003289 .00003311 .00003311 .00003205	

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CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,4,6,7,8-HpCDF

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501	Avian Egg	< .00025432	.00025432	< .00003268	.00003268	
shot9502	Avian Egg	< .00022694	.00022694	< .00003086	.00003086	
shot9503	Avian Egg	< .00031605	.00031605	< .00003268	.00003268	
shot9504	Avian Egg	< .00023259	.00023259	< .00003086	.00003086	
shot9505	Avian Egg	< .00025565	.00025565	< .00003247	.00003247	
shot9506	Avian Egg	< .0002722	.0002722	< .00003185	.00003185	
shot9507	Avian Egg	< .00027208	.00027208	< .00003311	.00003311	
shot9508	Avian Egg	< .00025426	.00025426	< .00003333	.00003333	28 ¹⁰¹
shot9509	Avian Egg	< .00024189	.00024189	< .00003268	.00003268	
shot9510	Avian Egg	< .00020343	.00020343	< .00003289	.00003289	
shot9511	Avian Egg	< .00021374	.00021374	< .00003247	.00003247	
shot9512	Avian Egg	< .00021642	.00021642	< .00003145	.00003145	
shot9513	Avian Egg	< .00020527	.00020527	< .00003268	.00003268	
shot9514	Avian Egg	< .00019136	.00019136	< .00003289	.00003289	
shot9515	Avian Egg	< .00021713	.00021713	< .00003289	.00003289	
shot9516	Avian Egg	< .00023139	.00023139	< .00003311	.00003311	
shot9517	Avian Egg	< .00021253	.00021253	< .00003311	.00003311	
	00					
shot9518	Avian Egg	< .00023515	.00023515	< .00003205	.00003205	

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CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,4,7,8,9-HpCDF

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501 shot9502 shot9503 shot9504 shot9505 shot9506 shot9506 shot9507 shot9508 shot9509 shot9510 shot9511 shot9512 shot9513 shot9514 shot9515 shot9516 shot9517	Avian Egg Avian Egg	<pre>< .00025432 < .00022694 < .00031605 < .00023259 < .00025565 < .0002722 < .00027208 < .00027208 < .00025426 < .00024189 < .00020343 < .00021374 < .00021374 < .00021642 < .00021642 < .00021527 < .00019136 < .00021713 < .00023139 < .00021253</pre>	.00025432 .00022694 .00031605 .00023259 .00025565 .0002722 .00027208 .00025426 .00025426 .00024189 .00020343 .00021374 .00021642 .00021642 .00020527 .00019136 .00021713 .00023139 .00021253	 < .00003268 < .00003086 < .00003268 < .00003268 < .00003247 < .00003247 < .00003311 < .00003268 < .00003268 < .00003289 < .00003247 < .00003247 < .00003289 < .00003211 	.00003268 .00003086 .00003268 .00003268 .00003247 .00003247 .00003311 .00003333 .00003268 .00003268 .00003289 .00003268 .00003289 .00003289 .00003289 .00003289 .00003289	
shot9512 shot9513 shot9514 shot9515 shot9516	Avian Egg Avian Egg Avian Egg Avian Egg Avian Egg	< .00021642 < .00020527 < .00019136 < .00021713 < .00023139	.00021642 .00020527 .00019136 .00021713 .00023139	< .00003145 < .00003268 < .00003289 < .00003289 < .00003311	.00003145 .00003268 .00003289 .00003289 .00003311	

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Lab Name: GERG

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CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,4,7,8-HxCDD

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501 shot9502 shot9503 shot9504 shot9505 shot9506 shot9507 shot9508 shot9509 shot9509 shot9510 shot9511 shot9512 shot9513 shot9515 shot9516 shot9517	Avian Egg Avian Egg	 < .00025432 < .00022694 < .00031605 < .00023259 < .00025565 < .0002722 < .00027208 < .00025426 < .00024189 < .00020343 < .00021374 < .00021642 < .00020527 < .00021713 < .00023139 < .00021253 	.00025432 .00022694 .00031605 .00023259 .00025565 .0002722 .00027208 .00025426 .00025426 .00024189 .00020343 .00021374 .00021642 .00020527 .00019136 .00021713 .00023139 .00021253	 < .00003268 < .00003086 < .00003268 < .00003268 < .00003247 < .00003185 < .00003311 < .00003268 < .00003268 < .00003247 < .00003247 < .00003247 < .00003248 < .00003248 < .00003248 < .00003289 < .00003281 < .00003281 < .00003281 < .00003281 < .00003281 < .00003311 < .00003311 	.00003268 .00003086 .00003268 .00003268 .00003247 .00003185 .00003311 .00003333 .00003268 .00003268 .00003247 .00003247 .00003268 .00003289 .00003289 .00003289 .00003311 .00003311	
shot9518	Avian Egg	< .00023515	.00023515	< .00003205	.00003205	

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CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,4,7,8-HxCDF

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501	Avian Egg	< .00025432	.00025432	< .00003268	.00003268	
shot9502	Avian Egg	< .00022694	.00022694	< .00003086	.00003086	
shot9503	Avian Egg	< .00031605	.00031605	< .00003268	.00003268	
shot9504	Avian Egg	< .00023259	.00023259	< .00003086	.00003086	
shot9505	Avian Egg	< .00025565	.00025565	< .00003247	.00003247	
shot9506	Avian Egg	< .0002722	.0002722	< .00003185	.00003185	
shot9507	Avian Egg	< .00027208	.00027208	< .00003311	.00003311	
shot9508	Avian Egg	< .00025426	.00025426	< .00003333	.00003333	8
shot9509	Avian Egg	< .00024189	.00024189	< .00003268	.00003268	
shot9510	Avian Egg	< .00020343	.00020343	< .00003289	.00003289	
shot9511	Avian Egg	< .00021374	.00021374	< .00003247	.00003247	
shot9512	Avian Egg	< .00021642	.00021642	< .00003145	.00003145	
shot9513	Avian Egg	< .00020527	.00020527	< .00003268	.00003268	
shot9514	Avian Egg	< .00019136	.00019136	< .00003289	.00003289	
shot9515	Avian Egg	< .00021713	.00021713	< .00003289	.00003289	
shot9516	Avian Egg	< .00023139	.00023139	< .00003311	.00003311	
shot9517	Avian Egg	< .00021253	.00021253	< .00003311	.00003311	
shot9518	Avian Egg	< .00023515	.00023515	< .00003205	.00003205	

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CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,6,7,8-HxCDD

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501 shot9502	Avian Egg Avian Egg	< .00025432 < .00022694	.00025432 .00022694	< .00003268 < .00003086	.00003268 .00003086	
shot9503 shot9504 shot9505	Avian Egg Avian Egg Avian Egg	< .00031605 < .00023259 < .00025565	.00031605 .00023259 .00025565	< .00003268 < .00003086 < .00003247	.00003268 .00003086 .00003247	
shot9506 shot9507	Avian Egg Avian Egg Avian Egg	< .00023303 < .0002722 < .00027208	.00023303	< .00003247 < .00003185 < .00003311	.00003247	Ŧ
shot9508 shot9509 shot9510	Avian Egg Avian Egg Avian Egg	< .00025426 < .00024189 < .00020343	.00025426 .00024189 .00020343	< .00003333 < .00003268 < .00003289	.00003333 .00003268 .00003289	
shot9511 shot9512	Avian Egg Avian Egg	< .00020343 < .00021374 < .00021642	.00020343	< .00003247 < .00003247 < .00003145	.00003247	
shot9513 shot9514	Avian Egg Avian Egg	< .00020527 < .00019136	.00020527	< .00003268 < .00003289	.00003268	
shot9515 shot9516 shot9517 shot9518	Avian Egg Avian Egg Avian Egg Avian Egg	< .00021713 < .00023139 < .00021253 < .00023515	.00021713 .00023139 .00021253 .00023515	< .00003289 < .00003311 < .00003311 < .00003205	.00003289 .00003311 .00003311 .00003205	

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CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,6,7,8-HxCDF

Samp Numb		Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
Numb Shot9 Sho	501Avian Egg502Avian Egg503Avian Egg504Avian Egg505Avian Egg506Avian Egg507Avian Egg508Avian Egg509Avian Egg510Avian Egg511Avian Egg512Avian Egg513Avian Egg514Avian Egg515Avian Egg516Avian Egg	<pre>(ppm Dry Weight) </pre> < .00025432 < .00022694 < .00031605 < .00023259 < .00025565 < .0002722 < .00027208 < .00025426 < .00024189 < .00020343 < .00021374 < .00021642 < .00021642 < .00021642 < .00021713 < .00023139 < .00021253	Limit .00025432 .00022694 .00031605 .00023259 .00025565 .0002722 .00027208 .00027208 .00027208 .00027208 .00027208 .00027208 .00027208 .00027208 .00027208 .00027208 .00027208 .00021374 .00021642 .00020527 .00019136 .00021713 .00023139 .00021253	<pre>(ppm Wet Weight) </pre> < .00003268 < .00003086 < .00003268 < .00003268 < .00003247 < .00003185 < .00003247 < .00003333 < .00003268 < .00003268 < .00003268 < .00003247 < .00003268 < .00003247 < .00003247 < .00003268 < .00003247 < .00003248 < .00003247 < .00003248 < .00003248 < .00003289 < .00003289 < .00003289 < .00003289 < .00003289 < .00003281 < .00003281	Limit .00003268 .00003086 .00003268 .00003268 .00003247 .00003247 .00003185 .00003247 .00003268 .00003268 .00003268 .00003268 .00003268 .00003268 .00003289 .00003289 .00003289	
shot95	00	< .00023515	.00023515	< .00003205	.00003205	

CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,7,8,9-HxCDD

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501	Avian Egg	< .00025432	.00025432	< .00003268	.00003268	
shot9502	Avian Egg	< .00022694	.00022694	< .00003086	.00003086	
shot9503	Avian Egg	< .00031605	.00031605	< .00003268	.00003268	
shot9504	Avian Egg	< .00023259	.00023259	< .00003086	.00003086	
shot9505	Avian Egg	< .00025565	.00025565	< .00003247	.00003247	
shot9506	Avian Egg	< .0002722	.0002722	< .00003185	.00003185	
shot9507	Avian Egg	< .00027208	.00027208	< .00003311	.00003311	
shot9508	Avian Egg	< .00025426	.00025426	< .00003333	.00003333	(1)
shot9509	Avian Egg	< .00024189	.00024189	< .00003268	.00003268	
shot9510	Avian Egg	< .00020343	.00020343	< .00003289	.00003289	
shot9511	Avian Egg	< .00021374	.00021374	< .00003247	.00003247	
shot9512	Avian Egg	< .00021642	.00021642	< .00003145	.00003145	
shot9513	Avian Egg	< .00020527	.00020527	< .00003268	.00003268	
shot9514	Avian Egg	< .00019136	.00019136	< .00003289	.00003289	
shot9515	Avian Egg	< .00021713	.00021713	< .00003289	.00003289	
shot9516	Avian Egg	< .00023139	.00023139	< .00003311	.00003311	
shot9517	Avian Egg	< .00021253	.00021253	< .00003311	.00003311	
shot9518	Avian Egg	< .00023515	.00023515	< .00003205	.00003205	

CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,7,8,9-HxCDF

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501 shot9502 shot9503 shot9504 shot9505 shot9506 shot9507 shot9508 shot9509 shot9510 shot9511	Avian Egg Avian Egg	<.00025432	.00025432 .00022694 .00031605 .00023259 .00025565 .0002722 .00027208 .00025426 .00025426 .00024189 .00020343 .00021374	<.00003268	.00003268 .00003086 .00003268 .00003268 .00003247 .00003247 .00003311 .00003333 .00003268 .00003289 .00003247	ŝ
shot9512 shot9513 shot9514 shot9515 shot9516 shot9517 shot9518	Avian Egg Avian Egg Avian Egg Avian Egg Avian Egg Avian Egg Avian Egg	<.00021642	.00021642 .00020527 .00019136 .00021713 .00023139 .00021253 .00023515	<.00003145 <.00003268 <.00003289 <.00003289 <.00003311 <.00003311 <.00003205	.00003145 .00003268 .00003289 .00003289 .00003311 .00003311 .00003205	

Catalog: 5050045

Lab Name: GERG 1/10/97

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CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,7,8-PeCDD

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501 shot9502 shot9503 shot9504 shot9505 shot9506 shot9507 shot9508 shot9509 shot9510 shot9511 shot9512 shot9513 shot9514 shot9515	Avian Egg Avian Egg	 < .00025432 < .00022694 < .00031605 < .00023259 < .00025565 < .0002722 < .00027208 < .00025426 < .00024189 < .00021374 < .00021374 < .00021642 < .00019136 < .00021713 	.00025432 .00022694 .00031605 .00023259 .00025565 .0002722 .00027208 .00025426 .00025426 .00024189 .00020343 .00021374 .00021642 .00020527 .00019136 .00021713	 < .00003268 < .00003086 < .00003268 < .00003268 < .00003247 < .00003185 < .00003311 < .00003268 < .00003268 < .00003289 < .00003247 < .00003247 < .00003248 < .00003248 < .00003248 < .00003268 < .00003268 < .00003289 	.00003268 .00003086 .00003268 .00003268 .00003247 .00003185 .00003311 .00003333 .00003268 .00003289 .00003247 .00003247 .00003268 .00003289 .00003289	14
shot9516 shot9517 shot9518	Avian Egg Avian Egg Avian Egg	< .00023139 < .00021253 < .00023515	.00023139 .00021253 .00023515	< .00003311 < .00003311 < .00003205	.00003311 .00003311 .00003205	
	00					

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CONTAMINANT CONCENTRATIONS

Analyte: 1,2,3,7,8-PeCDF

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501	Avian Egg	< .00025432	.00025432	< .00003268	.00003268	
shot9502	Avian Egg	< .00022694	.00022694	< .00003086	.00003086	
shot9503	Avian Egg	< .00031605	.00031605	< .00003268	.00003268	
shot9504	Avian Egg	< .00023259	.00023259	< .00003086	.00003086	
shot9505	Avian Egg	< .00025565	.00025565	< .00003247	.00003247	
shot9506	Avian Egg	< .0002722	.0002722	< .00003185	.00003185	
shot9507	Avian Egg	< .00027208	.00027208	< .00003311	.00003311	
shot9508	Avian Egg	< .00025426	.00025426	< .00003333	.00003333	
shot9509	Avian Egg	< .00024189	.00024189	< .00003268	.00003268	×.
shot9510	Avian Egg	< .00020343	.00020343	< .00003289	.00003289	
shot9511	Avian Egg	< .00021374	.00021374	< .00003247	.00003247	
shot9512	Avian Egg	< .00021642	.00021642	< .00003145	.00003145	
shot9513	Avian Egg	< .00020527	.00020527	< .00003268	.00003268	
shot9514	Avian Egg	< .00019136	.00019136	< .00003289	.00003289	
shot9515	Avian Egg	< .00021713	.00021713	< .00003289	.00003289	
shot9516	Avian Egg	< .00023139	.00023139	< .00003311	.00003311	
shot9517	Avian Egg	< .00021253	.00021253	< .00003311	.00003311	
shot9518	Avian Egg	< .00023515	.00023515	< .00003205	.00003205	
511010010	run Lgg	.00020010	.00020010	.00000200	.00000200	

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CONTAMINANT CONCENTRATIONS

Analyte: 2,3,4,6,7,8-HxCDF

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot950	1 Avian Egg	< .00025432	.00025432	< .00003268	.00003268	
shot950	2 Avian Egg	< .00022694	.00022694	< .00003086	.00003086	
shot950	3 Avian Egg	< .00031605	.00031605	< .00003268	.00003268	
shot9504	4 Avian Egg	< .00023259	.00023259	< .00003086	.00003086	
shot950	5 Avian Egg	< .00025565	.00025565	< .00003247	.00003247	
shot950	6 Avian Egg	< .0002722	.0002722	< .00003185	.00003185	
shot950	7 Avian Egg	< .00027208	.00027208	< .00003311	.00003311	
shot9508	B Avian Egg	< .00025426	.00025426	< .00003333	.00003333	
shot9509	Avian Egg	< .00024189	.00024189	< .00003268	.00003268	31
shot9510) Avian Egg	< .00020343	.00020343	< .00003289	.00003289	
shot951	1 Avian Egg	< .00021374	.00021374	< .00003247	.00003247	
shot9512	2 Avian Egg	< .00021642	.00021642	< .00003145	.00003145	
shot9513	00	< .00020527	.00020527	< .00003268	.00003268	
shot9514		< .00019136	.00019136	< .00003289	.00003289	
shot9515	the second se	< .00021713	.00021713	< .00003289	.00003289	
shot9516		< .00023139	.00023139	< .00003311	.00003311	
shot9517	00	< .00021253	.00021253	< .00003311	.00003311	
shot9518		< .00023515	.00023515	< .00003205	.00003205	

CONTAMINANT CONCENTRATIONS

Analyte: 2,3,4,7,8-PeCDF

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501	Avian Egg	< .00025432	.00025432	< .00003268	.00003268	
shot9502	Avian Egg	< .00022694	.00022694	< .00003086	.00003086	
shot9503	Avian Egg	< .00031605	.00031605	< .00003268	.00003268	
shot9504	Avian Egg	< .00023259	.00023259	< .00003086	.00003086	
shot9505	Avian Egg	< .00025565	.00025565	< .00003247	.00003247	
shot9506	Avian Egg	< .0002722	.0002722	< .00003185	.00003185	
shot9507	Avian Egg	< .00027208	.00027208	< .00003311	.00003311	
shot9508	Avian Egg	< .00025426	.00025426	< .00003333	.00003333	
shot9509	Avian Egg	< .00024189	.00024189	< .00003268	.00003268	54
shot9510	Avian Egg	< .00020343	.00020343	< .00003289	.00003289	
shot9511	Avian Egg	< .00021374	.00021374	< .00003247	.00003247	
shot9512	Avian Egg	< .00021642	.00021642	< .00003145	.00003145	
shot9513	Avian Egg	< .00020527	.00020527	< .00003268	.00003268	
shot9514	Avian Egg	< .00019136	.00019136	< .00003289	.00003289	
shot9515	Avian Egg	< .00021713	.00021713	< .00003289	.00003289	
shot9516	Avian Egg	< .00023139	.00023139	< .00003311	.00003311	
shot9517	Avian Egg	< .00021253	.00021253	< .00003311	.00003311	
shot9518	Avian Egg	< .00023515	.00023515	< .00003205	.00003205	
011010010			.00020010	.00000200		

CONTAMINANT CONCENTRATIONS

Analyte: 2,3,7,8-TCDD

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501	Avian Egg	.00047082	.00005086	.0000605	.00000654	
shot9502	Avian Egg	.00032426	.00004539	.0000441	.00000617	
shot9503	Avian Egg	.00103482	.00006321	.000107	.00000654	
shot9504	Avian Egg	.00085154	.00004652	.000113	.00000617	
shot9505	Avian Egg	.00055118	.00005113	.00007	.00000649	
shot9506	Avian Egg	.00137607	.00005444	.000161	.00000637	
shot9507	Avian Egg	.00038209	.00005442	.0000465	.00000662	
shot9508	Avian Egg	.00045919	.00005085	.0000602	.00000667	
shot9509	Avian Egg	.0000792	.00004838	.0000107	.00000654	
shot9510	Avian Egg	.0005368	.00004069	.0000868	.00000658	
shot9511	Avian Egg	< .00004275	.00004275	< .00000649	.00000649	
shot9512	Avian Egg	.00004708	.00004328	.00000684	.00000629	
shot9513	Avian Egg	.00006972	.00004105	.0000111	.00000654	
shot9514	Avian Egg	.00014776	.00003827	.0000254	.00000658	
shot9515	Avian Egg	.00015842	.00004343	.000024	.00000658	
shot9516	Avian Egg	.00007687	.00004628	.000011	.00000662	
shot9517	Avian Egg	.00024583	.00004251	.0000383	.00000662	
shot9518	Avian Egg	< .00004703	.00004703	< .00000641	.00000641	

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CONTAMINANT CONCENTRATIONS

Analyte: 2,3,7,8-TCDF

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501	Avian Egg	< .00005086	.00005086	< .00000654	.00000654	
shot9502	Avian Egg	< .00004539	.00004539	< .00000617	.00000617	
shot9503	Avian Egg	< .00006321	.00006321	< .00000654	.00000654	
shot9504	Avian Egg	< .00004652	.00004652	< .00000617	.00000617	
shot9505	Avian Egg	< .00005113	.00005113	< .00000649	.00000649	
shot9506	Avian Egg	< .00005444	.00005444	< .00000637	.00000637	
shot9507	Avian Egg	< .00005442	.00005442	< .00000662	.00000662	
shot9508	Avian Egg	< .00005085	.00005085	< .00000667	.00000667	
shot9509	Avian Egg	< .00004838	.00004838	< .00000654	.00000654	
shot9510	Avian Egg	< .00004069	.00004069	< .00000658	.00000658	
shot9511	Avian Egg	< .00004275	.00004275	< .00000649	.00000649	
shot9512	Avian Egg	< .00004328	.00004328	< .00000629	.00000629	
shot9513	Avian Egg	< .00004105	.00004105	< .00000654	.00000654	
shot9514	Avian Egg	< .00003827	.00003827	< .00000658	.00000658	
shot9515	Avian Egg	< .00004343	.00004343	< .00000658	.00000658	
shot9516	Avian Egg	< .00004628	.00004628	< .00000662	.00000662	
shot9517	Avian Egg	< .00004251	.00004251	< .00000662	.00000662	
shot9518	Avian Egg	< .00004703	.00004703	< .00000641	.00000641	

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CONTAMINANT CONCENTRATIONS

Analyte: OCDD

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
 shot9501	Avian Egg	< .00050863	.00050863	< .00006536	.00006536	
shot9502	Avian Egg	< .00045389	.00045389	< .00006173	.00006173	
shot9503	Avian Egg	< .0006321	.0006321	< .00006536	.00006536	
shot9504	Avian Egg	< .00046517	.00046517	< .00006173	.00006173	
shot9505	Avian Egg	< .0005113	.0005113	< .00006494	.00006494	
shot9506	Avian Egg	< .0005444	.0005444	< .00006369	.00006369	
shot9507	Avian Egg	< .00054417	.00054417	< .00006623	.00006623	
shot9508	Avian Egg	< .00050852	.00050852	< .00006667	.00006667	
shot9509	Avian Egg	< .00048379	.00048379	< .00006536	.00006536	×
shot9510	Avian Egg	< .00040686	.00040686	< .00006579	.00006579	
shot9511	Avian Egg	< .00042749	.00042749	< .00006494	.00006494	
shot9512	Avian Egg	< .00043285	.00043285	< .00006289	.00006289	
shot9513	Avian Egg	.0004598	.00041055	.0000732	.00006536	
shot9514	Avian Egg	.00049506	.00038272	.0000851	.00006579	
shot9515	Avian Egg	< .00043425	.00043425	< .00006579	.00006579	
shot9516	Avian Egg	< .00046279	.00046279	< .00006623	.00006623	
shot9517	Avian Egg	< .00042507	.00042507	< .00006623	.00006623	
shot9518	Avian Egg	< .0004703	.0004703	< .0000641	.0000641	

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CONTAMINANT CONCENTRATIONS

Analyte: OCDF

Sample Number	Sample Matrix	Result (ppm Dry Weight)	Detection Limit	Result (ppm Wet Weight)	Detection Limit	
shot9501	Avian Egg	< .00050863	.00050863	< .00006536	.00006536	
shot9502 shot9503 shot9504	Avian Egg Avian Egg Avian Egg	< .00045389 < .0006321 < .00046517	.00045389 .0006321 .00046517	< .00006173 < .00006536 < .00006173	.00006173 .00006536 .00006173	
shot9505 shot9506	Avian Egg Avian Egg	< .00040317 < .0005113 < .0005444	.0005113	< .00006494 < .00006369	.00006494	
shot9507 shot9508	Avian Egg Avian Egg	< .00054417 < .00050852	.00054417	< .00006623 < .00006623 < .00006667	.00006623	
shot9509 shot9510	Avian Egg Avian Egg	< .00048379	.00048379	< .00006536	.00006536	
shot9511 shot9512	Avian Egg Avian Egg	< .00042749 < .00043285	.00042749	< .00006494 < .00006289	.00006494	
shot9513 shot9514	Avian Egg Avian Egg	< .00041055 < .00038272	.00041055	< .00006536 < .00006579	.00006536	
shot9515 shot9516 shot9517 shot9518	Avian Egg Avian Egg Avian Egg Avian Egg	< .00043425 < .00046279 < .00042507 < .0004703	.00043425 .00046279 .00042507 .0004703	< .00006579 < .00006623 < .00006623 < .0000641	.00006579 .00006623 .00006623 .0000641	

Appendix B. Analytical methods used for the analysis of 2,3,7,8 TCDD in bird eggs collected from Shooter's Island and the Isle of Meadows, New York Bight, New York/New Jersey.

LABORATORY: Geochemical & Environmental Research Group, Texas A&M

% Dry Weight

Approximately 1 gram of wet sample is weighed into a clean, labeled, preweighed 10 ml beaker. The beaker is placed in a forced air oven at approximately 75 degrees Celsius for 24 hours. The beaker with the dry sample is then weighed and the % dry weight is calculated by the formula:

(wt. dry sample and beaker) - (wt. beaker) (100)

(wt. wet sample and beaker) - (wt. beaker)

Tissue Organics

I. The tissue samples were extracted by the NOAA Status and Trends Method (MacLeod et al., 1985) with minor revisions (Brooks et al., 1989; Wade et al., 1988). Briefly, the tissue samples were homogenized with a Teckmar Tissumizer. A 1 to 10-gram sample (wet weight) was extracted with the Teckmar Tissumizer by adding surrogate standards, Na2SO4, and methylene chloride in a centrifuge tube. The tissue extracts were purified by silica/alumina column chromatography to isolate the aliphatic and PAH/pesticide/PCB fractions. The PAH/pesticide/PCB fraction was further purified by HPLC in order to remove interfering lipids.

The quantitative analyses were performed by capillary gas chromatography (CGC) with a flame ionization detector for aliphatic hydrocarbons, CGC with electron capture detector for pesticides and PCB's, and a mass spectrometer detector in the SIM mode for aromatic hydrocarbons (Wade et al., 1988).

There are specific cases where analytes requested for the pesticide and PCB analyses and are known to co-elute with other analytes in the normal CGC with electron capture. These include the pesticide Endosulfan I and the PCB congeners 114 and 157. In these cases, the samples will be analyzed by CGC with a mass spectrometer detector in the SIM mode.

References

- Brooks, J.M., T.L. Wade, E.L. Atlas, M.C. Kennicutt II, B.J. Presley, R.R. Fay, E.N. Powell, and G. Wolff (1989) Analysis of Bivalves and Sediments for Organic Chemicals and Trace Elements.
- MacLeod, W.D., D.W. Brown, A.J. Friedman, D.G. Burrow, O. Mayes, R.W. Pearce, C.A. Wigren, and R.G. Bogar (1985) Standard Analytical Procedures of the NOAA National Analytical Facility 1985-1986. Extractable Toxic Organic Compounds. 2nd Ed. U.S. Department of Commerce, NOAA/NMFS, NOAA Tech. Memo. NMFS F/NWRC-92.
- Wade, T.L., E.L. Atlas, J.M. Brooks, M.C. Kennicutt II, R.G. Fox, J. Sericano, B. Garcia, and D. DeFreitas (1988) NOAA Gulf of Mexico Status and Trends Program: Trace Organic Contaminant Distribution in Sediments and Oyster. Estuaries 11, 171-179.

Dioxin/Furan Analysis

XXVI. The procedure uses matrix specific extraction, analyte specific cleanup, and HRGC/HRMS analysis techniques. If interferences are encountered, the method provides selected cleanup procedures to aid in their elimination.

A specified amount of the sample matrix is spiked with a solution containing each of fifteen isotopically (13C12) labeled PCDDs/PCDFs. The sample is then extracted according to a matrix specific extraction procedure. Aqueous samples that are judged to contain 1% or more solids, and solid samples that show an aqueous phase, are filtered, the solid phase and the aqueous phase extracted separately, and the extracts combined before cleanup. Following a solvent exchange step, the extracts are cleaned up by column chromatography on alumina, silica gel, and AX-21 activated carbon on silica. The preparation of the final extract for HRGA/HRMS analysis is accomplished by adding two isotopically (13C12) labeled recovery standards.

Two uL of the concentrated extracts are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10% valley definition). The identification of the sixteen 2378-substituted isomers for which a 13C-labeled standard is available is based on their elution at their exact retention time and the simultaneous detection of the two most abundant ions in the molecular ion region. The identification of OCDF is based on its retention time relative to 13C-OCDD. Confirmation is based on a comparison of the ratios of the integrated ion abundance of the molecular ion species to their theoretical abundance ratios. Quantitation of the individual congeners is achieved in conjunction with the establishment of a multipoint calibration curve for each homologue, during which each calibration solution is analyzed once.

REFERENCES

Tondeur, Yves, "Method 8290: Analytical Procedures and Quality Assurance for Multimedia Analysis of Polychlorinated Dibenzo-p-dioxins and Dibenzofurans by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry", USEPA EMSL, Las Vegas, Nevada, June 1987. (Revision 0, November 1990.)

USEPA Office of Water Regulation and Standards, Industrial Technology Division, "Method 1613: Tetra- through Octa- Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS", Revision A, April 1990.