DETERMINATION AND COMPARISON OF PERSISTENT ORGANOCHLORINE COMPOUNDS IN THE GREAT HORNED OWL (*Bubo virgianius*) – LIVER VERSUS WHOLE CARCASS

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Introduction

Great horned owls were collected from the Denver, Colorado area and analyzed to determine the burden of polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), co-planar polychlorinated biphenyls (Co-PCBs) and mono-ortho polychlorinated biphenyls (MO-PCBs). This species was selected as a monitor for persistent organochlorine compounds because they are commonly found in the region and they feed within the nocturnal food web. Collection of the animals occurred from 1996 to 1998. Samples were stored frozen and supplied to the laboratory for analysis between 1998 and 2000.

Initially only liver samples were provided to the laboratory. The liver samples were analyzed for PCDD/PCDF and Co-PCBs in 1998. Analysis of eight whole carcass owls for PCDD/PCDF Co-PCBs, and MO-PCBs was conducted in 1999. To provide a comparison with the data for the whole carcass owls, extracts were analyzed for MO-PCBs in 2000. Results for the analyses are presented in Table 1.

Materials and Methods

All samples were prepared and analyzed according to isotope dilution or internal standard quantitation based on the techniques presented in US EPA Method 8290[1], 1613B[2], and Method 1668[3]. The MO-PCB analysis of the liver samples was conducted using an internal standard approach on a separate extract.

All samples were dried and fortified with a mixture of ^{13}C-labeled PCDD/PCDF/PCB internal quantitation standards (IQS) or in the case of the MO-PCBs from livers, PCB 30 internal standard. The samples and quality control (QC) samples were then extracted by soxhlet for 16 hours. The extracts were then concentrated and their percent lipid was measured. The extracts were fortified with ^{37}Cl labeled 2,3,7,8-TCDD as a clean-up efficiency standard. The samples were then subjected to an acid silica slurry to remove bulk lipids followed by a silica column. The whole carcass owl extracts were then split 1:1 for MO-PCB analysis and PCDD/PCDF/Co-PCB analysis. The latter split was also taken through a neutral alumina column followed by an FMS® carbon column. The extracts were all concentrated and fortified with a ^{13}C-labeled recovery standard. The final volumes were 10 µL for PCDD/PCDF/Co-PCB splits and 20 µL for the MO-PCB splits.

HRGC/HRMS was employed for sample analysis using a Hewlett-Packard 5890 gas chromatograph interfaced to a VG-70S-250 double focusing/dual sector high resolution mass spectrometer. A DB-5MS gas chromatography column (60 meter, 0.25 mm ID, 0.25 μm film thickness) was used. Mass spectrometer acquisition was performed at 10,000 resolution in the selected ion monitoring mode (SIM). A six-point initial calibration curve (I-CAL) for PCDD/PCDF was performed using a 0.25-200 pg/μL range for tetra-substituted isomers. Co-PCB and MO-PCB I-CAL was also a six-point curve using a 2.5-500 pg/μL range for all isomers. Each standard conformed to a method criteria of <20% RSD (natives) and <30% RSD (^{13}C) from the I-CAL. Data acquisition was performed simultaneously for PCDD/PCDF and Co-PCB isomers from a single injection.

Results and Discussion

The results in Table 1 compare native analyte concentrations for the livers and carcasses. The concentrations are based on lipid weight for all samples, except the owl livers for MO-PCBs. These were based on wet-weight. Method Blank results are given at the end of the table. LCS results are not given here for reasons of brevity, but all passed within 75-125% for native analyte recovery. Calculations for %contribution to I-TEQ[4] given at the bottom of Table 1 used detected results and noise-based detection limits for non-detects.

The data shows there are higher levels of PCDD/PCDF and Co-PCBs in the livers of three samples (i.e., 96FGH007, 96FGH017, and 96FGH002) than there are in the whole carcass owls. This general trend seems to be reversed, however, for the MO-PCBs again with the exception of two samples (i.e., 96FGH017 and 96FGH002).

The differences between these results are being investigated. One explanation for this reversed trend in the three samples (i.e. all low whole carcass mass) might be that emaciation of an animal can often lead to a migration of persistent organochlorine compounds out of the adipose tissue and into secondary organs[5].

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References

1. USEPA Method 1613, "Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, Revision B." Oct. 1994.
2. USEPA Method 8290, "Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography / High Resolution Mass Spectrometry (HRGC/HRMS), Revision 0." September 1994.
3. Draft Method 1668 for the Measurement of Toxic PCB Congeners by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry. Draft Revision. March, 1997
4. World Health Organization Meeting in Stockholm, Sweden, 15-18 June, 1997 (Van den Berg et al., 1998).
5. Hoffman, D.J., C.P. Rice, and T.J. Kubiak, PCBs and Dioxins in Birds, Environmental Contaminants in Wildlife: Interpreting Tissue Residues, 1996, pp. 165-207. Lewis Publishers.

Table 1. Results Summary for Whole Owls vs. Owl Livers (pg/g)

	96RFGH03		96RFGH07		96RFGH01		96FGH007		96RFGH12	
PCDD/PCDF	Whole Owl	Liver	Whole Owl	Liver	Whole Owl	Liver	Whole Owl	Liver	Whole Owl	Liver
2378-TCDF	LT	8.72	LT	0.379	LT	5.73	LT	4.26	LT	LT
2378-TCDD	LT	LT	0.870	LT	0.698	LT	3.90	10.8	LT	0.675
12378-PeCDF	4.18	LT	2.94	0.176	2.58	LT	12.8	LT	4.81	LT
23478-PeCDF	14.3	68.1	5.67	5.66	2.29	LT	53.1	1352	5.32	14.2
12378-PeCDD	6.74	LT	6.85	3.15	3.01	LT	32.1	254	5.01	4.72
123478-HxCDF	22.6	252	10.1	9.20	4.60	LT	86.7	4190	10.0	16.3
123678-HxCDF	LT	74.0	LT	4.69	LT	LT	LT	2004	LT	7.16
234678-HxCDF	LT	22.7	LT	LT	LT	LT	11.6	363	2.91	LT
123789-HxCDF	3.33	LT	LT	LT	1.20	3.63	5.19	86.3	2.42	1.36
123478-HxCDD	10.0	38.2	6.31	5.97	1.36	1.82	33.3	595	4.33	7.97
123678-HxCDD	19.3	78.9	15.3	13.4	2.89	2.92	84.2	1724	7.89	22.4
123789-HxCDD	2.65	LT	1.32	LT	LT	LT	6.36	146	2.10	LT
1234789-HpCDF	LT	16.5	LT	LT	LT	LT	LT	350	LT	LT
1234678-HpCDD	53.9	417	27.6	26.0	1.46	LT	75.9	2396	6.97	25.1
OCDF	44.3	19.6	23.3	4.63	32.9	LT	60.1	143	118	5.24
OCDD	34.6	128	84.5	21.0	4.46	21.9	57.4	1107	14.8	16.4
Co-Planar PCBs										
PCB-81	LT	LT	2.18	0.421	2.60	15.9	1.23	LT	2.33	0.955
PCB-77	42.5	23.7	28.9	2.54	37.5	LT	60.3	21.4	32.5	6.87
PCB-126	54.7	128	42.0	28.5	69.6	43.3	314	4066	29.6	30.2
PCB-169	32.8	178	37.0	60.0	45.1	61.9	126	4041	44.6	245
Mono-Ortho PCBs										
PCB-123	132	LT	131	LT	147	LT	1380	769	66.6	LT
PCB-118	10200	1690	10700	1020	14400	1090	S	85400	6910	830
PCB-114	257	LT	253	LT	314	LT	8920	7260	183	LT
PCB-105	2910	401	2840	255	4100	273	35300	15000	2350	313
PCB-167	1250	173	1380	LT	1810	79.8	9900	3650	903	72.0
PCB-156	2470	251	4010	200	4740	179	91500	33500	5000	326
PCB-157	493	LT	805	41.3	852	36.0	20000	4930	944	60.4
PCB-189	494	LT	789	28.8	1070	LT	8780	1250	630	27.6
% of I-TEQ for PCBs	23.6	13.2	22.1	25.4	60.0	39.2	41.3	20.2	30.9	22.9

LT - Not detected based on noise based detection limits or Estimated Maximum Possible Concentration due to incorrect ratio or diphenyl ether interference

S - Peak saturated

Table 1. Results Summary for Whole Owls vs. Owl Livers (pg/g) (continued)

PCDD/PCDF	96FGH017		96RFGH05		96FGH002		Whole Owl Blank	Liver Blank 1	Liver Blank 2
	Whole Owl	Liver	Whole Owl	Liver	Whole Owl	Liver			
2378-TCDF	LT	1.61	LT	0.503	LT	3.54	LT	2.46	3.18
2378-TCDD	1.88	10.6	LT	0.919	1.75	12.5	LT	LT	LT
12378-PeCDF	3.86	LT	2.00	LT	2.06	14.3	0.696	LT	3.27
23478-PeCDF	6.20	242	3.01	4.17	3.59	164	LT	LT	LT
12378-PeCDD	12.7	182	3.78	5.46	6.89	120	LT	LT	3.94
123478-HxCDF	16.8	871	6.43	3.10	8.60	529	1.84	LT	1.39
123678-HxCDF	LT	386	LT	1.53	LT	124	1.39	LT	LT
234678-HxCDF	LT	69.1	1.74	LT	LT	54.0	0.599	LT	1.68
123789-HxCDF	1.79	14.2	LT	0.778	LT	3.26	1.07	LT	LT
123478-HxCDD	6.09	181	1.93	3.74	4.10	160	LT	LT	3.44
123678-HxCDD	18.2	678	4.67	9.84	12.1	663	0.578	LT	LT
123789-HxCDD	2.62	52.6	LT	0.648	1.98	74.5	LT	LT	LT
1234789-HpCDF	LT	41.0	LT	LT	LT	LT	1.71	LT	LT
1234678-HpCDD	10.6	442	LT	5.89	14.9	1116	8.36	3.64	LT
OCDF	51.9	19.9	75.8	LT	26.0	18.8	20.0	LT	LT
OCDD	19.1	187	8.62	7.56	26.1	487	30.7	15.0	22.6
Co-Planar PCBs									
PCB-81	3.32	LT	2.36	LT	2.35	LT	LT	LT	71.9
PCB-77	54.0	11.4	29.6	5.65	74.4	42.9	6.71	14.8	LT
PCB-126	74.3	2093	48.7	92.8	89.6	3981	LT	LT	4.27
PCB-169	29.6	1878	77.7	238	22.4	2134	LT	0.871	LT
Mono-Ortho PCBs									
PCB-123	212	197	159	124	365	1260	LT	*LT	LT
PCB-118	26500	40400	13600	11200	33700	160000	346	*22.7	*18.4
PCB-114	914	1900	333	LT	729	5010	LT	*LT	*LT
PCB-105	9570	12000	3240	2380	10100	37800	108	*15.9	*11.8
PCB-167	1910	3260	2610	1680	3000	11500	LT	*LT	*2.71
PCB-156	9890	15100	6400	3410	7830	24300	12.2	*LT	*10.2
PCB-157	1930	2740	1150	602	1670	4310	2.27	*LT	*3.12
PCB-189	1140	2010	1420	524	1290	2690	LT	*LT	*3.14
% of I-TEQ for PCBs	38.3	30.7	47.6	57.3	50.9	53.6	7.63	8.98	3.40

* These blank results were taken from a separate blank than the results given above them

% of I-TEQ for PCBs is the % contribution of the total PCBs compared to the total I-TEQ. I-TEFs were taken from WHO meeting, 1997, for Humans/Mammals[3]