#### Chlordane residue levels and geographic distribution of chlordane in fish from the non-tidal portion of the Patuxent River

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U. S. Fish and Wildlife Service Environmental Contaminants Branch Annapolis Field Office Annapolis, MD 21401

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#### EXECUTIVE SUMMARY

Chlordane is a broad-spectrum organochlorine insecticide widely used in the United States from the 1940s to the 1970s to control termites and agricultural, home, and garden insect pests. In the early 1970s, evidence arose that chlordane affected not only these targeted insect pests, but non-target organisms as well. Chlordane was found to be moderately toxic to mammals, slightly toxic to birds, and highly toxic to fishes, lower aquatic organisms, and beneficial insects. In addition, chlordane tends to bioaccumulate in the body fat, liver, and kidneys of organisms where it may be metabolized into more toxic compounds.

In 1974 the U.S. Environmental Protection Agency (EPA) issued a notice to cancel certain registered uses of chlordane because of its suspected carcinogenicity and other environmental concerns. In 1975 the EPA suspended these uses of chlordane after receiving further evidence of its carcinogenicity. On October 1, 1987, the State of Maryland restricted the sale of chlordane to certified pesticide applicators in an effort to avoid further dissemination of this already ubiquitous compound throughout the environment.

Since 1977, Maryland Department of the Environment (MDE) has been conducting annual statewide fish tissue analyses for organic compounds, including chlordane, as part of the federally-mandated Basic Water Monitoring Program. Results of these analyses have indicated a temporal variation pattern in chlordane levels in fish from non-tidal areas. Data from the Patuxent River, which is included in MDE's surveys, have shown that chlordane levels in fish from the non-tidal portion of the river tend to have peaked in 1983-84.

The objectives of the current study were:

- (1) to determine chlordane levels in fish found in the Patuxent River and compare these with State data; and
- (2) to determine the geographic distribution of chlordane present in fish from the coastal plain non-tidal portion of the Patuxent River.

Results of this study showed that chlordane residue levels in fish were lower than those found in similar areas of the Patuxent River in 1983-84. Decreasing chlordane levels from upstream to downstream areas was noted, but the trend proved not to be significant.

The data from this study tend to support MDE's findings that chlordane levels in the non-tidal portion of the Patuxent River appear to have peaked in 1983-84. Extreme variability in the results indicated that the geographic distribution of chlordane levels did not represent a significantly linear relationship despite the noted decreasing trend. That this trend proved not to be significant may have been due to the small sample size involved or the proximity of the sample stations. Larger sample sizes and sampling stations farther apart would help define if a

Title: Chlordane residue concentrations and geographic distribution of chlordane in fish from the non-tidal portion of the Patuxent River.

Abstract: As part of the federally-mandated Basic Water Monitoring Program, Maryland Department of the Environment (MDE) has been conducting annual statewide fish tissue analyses for organic compounds, including chlordane. Results have shown a temporal variation pattern in chlordane residue levels in fish from non-tidal areas since 1977. The Patuxent River is one the rivers included in this survey. The objectives of this study were: (1) to determine chlordane levels in fish found in the Patuxent River and compare these with State data; and (2) determine the geographic distribution of chlordane present in fish from the coastal plain non-tidal portion of the Patuxent River.

Three brown bullheads\* and a sediment sample were collected from each of six locations on the Patuxent and Little Patuxent Rivers from Laurel, Maryland to Davidsonville, Maryland. One of the fish specimens from each site was separated into fillet and carcass portions for analysis in order to determine chlordane residue levels in edible portions. The remaining two specimens underwent whole body analysis. Samples were subjected to an organochlorine scan to detect chlordane residue levels.

All but one of the fish specimens had detectable levels of chlordane, however these levels were lower than those found by MDE in the same areas in 1983-84. None of the sediment samples had detectable chlordane levels. A trend of decreasing levels in fish was noted from upstream to downstream, but the difference was not significant.

The lower chlordane levels in fish found in this section of the Patuxent River in 1988 tend to support the temporal variation pattern previously noted by MDE. Chlordane levels appear to have peaked in 1983-84. The fact that the noted decreasing trend in chlordane levels from upstream to downstream stations was not significant may have been due to small sample size or sample site proximity. There does not appear to be a health risk associated with chlordane levels in fish in this section of the Patuxent River at the present time.

Key words: chlordane, Patuxent River, Little Patuxent River, MDE, non-tidal, brown bullhead

\* At one station only one brown bullhead was caught. The remaining two specimens from this site were white catfish.

#### **ACKNOWLEDGEMENTS**

The study design and field sample collection for this study were conducted by U.S. Fish and Wildlife Service (FWS) contaminant biologist Daniel J. Audet, formerly with the Annapolis Field Office, now with the Chesapeake Bay Estuary Program\*. The statistical analyses were performed by David Scott, formerly with the Chesapeake Bay Estuary Program, now with the Ohio Division of Wildlife\*\*. Their efforts are greatly appreciated. We would also like to thank Dan Audet for his review and editing of this report.

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## TABLE OF CONTENTS

																					Page
Executive Summary .																	•				i
Abstract					٠								٠		•						iii
Acknowledgements .						٠		٠					•	٠		•					iv
Table of Contents .						٠	٠	•		٠											v
List of Tables																					vi
List of Figures																					vii
List of Appendices																					viii
Introduction																					1
Methods	si:	s.	:	:	•				•	:	:	•	:	:		•	•	:	:	:	1 1 3 5
Results																					6
Discussion																					6
Conclusions and Rec	comi	nei	nda	at:	io	ns															13
Literature Cited .						٠															14
Appendices																					16

## LIST OF TABLES

	<u>Pa</u>	ge
Table 1.	Compounds included in the organochlorine scan	4
Table 2.	Chlordane residue levels (ppm wet weight concentration) found in brown bullheads collected from the non-tidal portion of the Patuxent River	7

# LIST OF FIGURES

		<u>Page</u>
Figure 1.	General location of study area and sampling	
	stations	. 2

# LIST OF APPENDICES

			Page
Appendix	Α.	Lengths and weights of Patuxent River samples submitted for chlordane analysis	16
Appendix	В.	Analytical results of chemical analysis of fish and sediment samples from the Patuxent River	19

#### INTRODUCTION

Since 1977, the Maryland Department of the Environment (MDE) (formerly Maryland Office of Environmental Programs) has been conducting a statewide fish tissue analysis for organic compounds, including chlordane, as part of a Federally mandated Basic Water Monitoring Program (BWMP). Chlordane is toxic to various organisms, especially aquatic organisms, and poses a potential human health risk. In light of this concern the Food and Drug Administration (FDA) established an action level for chlordane in fish fillets of 0.3 parts per million (ppm) wet weight. Fish with elevated chlordane levels, x > 0.3 ppm, have been reported in several rivers in Maryland. Chlordane levels as high as 0.8840 ppm for whole fish have been reported from bluegill (Lepomis macrochirus) and 0.415 ppm for fillets from white sucker (Catostomus commersoni) caught in the freshwater portion of the Patuxent River (Murphy 1986). Other fish species analyzed from the Patuxent River with high levels of chlordane include brown bullhead (Ictalurus nebulosus) and fallfish (Semotilus corporalis). While these high levels are well documented, there is little understanding of the extent and geographic distribution of chlordane bioaccumulation in fish of the Patuxent River.

The objectives of the present study were as follows:

- to determine levels of chlordane present in fish found in the Patuxent River and compare with State data; and
- analyze the geographic distribution of chlordane present in fish of the coastal plain non-tidal portion of the Patuxent River.

#### MATERIALS AND METHODS

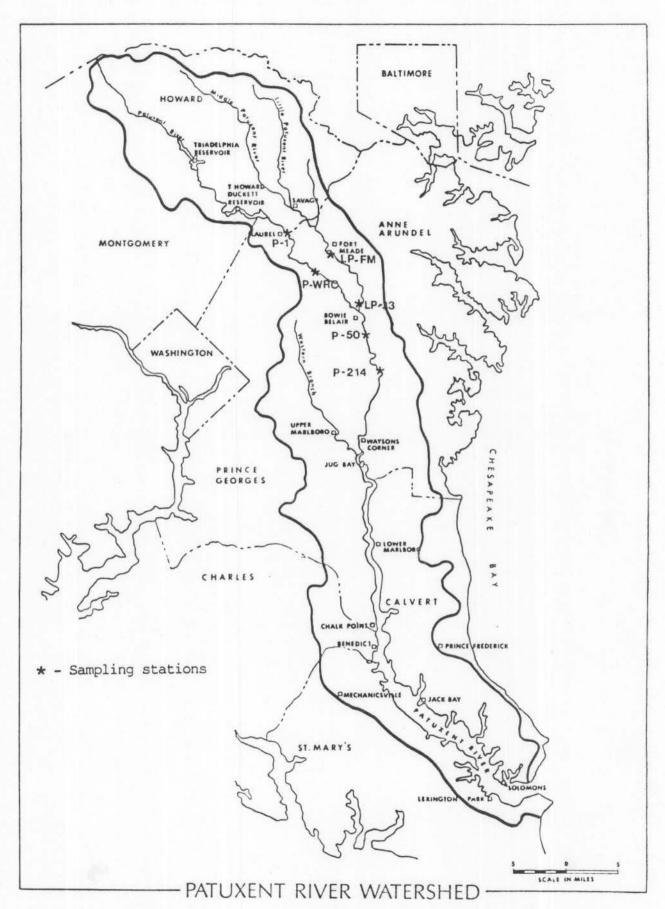
#### Sampling

10

Six stations within the non-tidal portion of the Patuxent and Little Patuxent Rivers were sampled between April 15 and June 23, 1988 (Figure 1):

- 1. the Patuxent River off of Route 1 in Laurel (P-1);
- the Patuxent River near Duvall Bridge at the Patuxent Wildlife Research Center (P-WRC);
- 3. the Patuxent just upstream of the Route 50 Bridge (P-50);
- the Patuxent River just upstream of the Route 214 Bridge (P-214);
- the Little Patuxent River just upstream of the Priest Bridge at Route 3 (LP-3); and

11



<sup>\*</sup> From "Patuxent River Commission Progress Report: 1980-1986", Maryland Department of State Planning, 1987.

 the Little Patuxent River near Route 424 at Fort Meade (LP-FM).

Sampling stations were selected based on MDE's study design. Station P-1 corresponds to MDE's site PXT0808, P-WRC corresponds to PXT0728, LP-3 corresponds to PXT0630, and P-50 corresponds to PXT0603.

MDE data (Butler and Allison 1984, Murphy 1986, and Murphy 1988) suggests that the brown bullhead has one of the most widespread distributions in the freshwater portion of the Patuxent River. In addition, brown bullheads have been found with high levels of chlordane in the Patuxent River. For these reasons, the brown bullhead was selected as the main species in this study. Access to the sample sites was by cance. Fish were collected by a combination of catfish traps, hoop nets (length: 4 feet; mesh size: 2 inches), gill net (experimental with mesh sizes ranging from 1.5 to 4 inches), and electroshocking with a backpack unit (Smith and Root). Collection techniques were dependent on varying conditions at each station.

Three brown bullheads were collected from each of the first five sampling stations for contaminant analysis between April 15 and June 23, 1988. Due to an inability to collect three bullheads from the sixth station (LP-FM), two white catfish (Ictalurus catus) and one brown bullhead were collected and submitted for analysis.

Composited sediment samples were collected from each of the six stations at areas of sediment deposition and analyzed for organochlorine pesticides, including chlordane. Three grabs per sample were made with a petite Ponar dredge (sampling area inlet is 6 X 6 inches).

#### Analysis

12

Two of the three fish specimens collected at each station were analyzed for whole body burden of organochlorines, while the remaining fish was separated into fillet and carcass portions prior to analysis. This approach allowed for a comparison of the two analytical procedures and a comparison of the results with State data. All samples were sent to the Mississippi State Chemical Laboratory (MSCL), Box CR, Mississippi State, Mississippi, 39762, for organochlorine analysis. The compounds included in the organochlorine scan are presented in Table 1. Analytical techniques used by MSCL are checked and approved by the U.S. Fish and Wildlife Service's (FWS) Patuxent Analytical Control Facility (PACF), which is responsible for quality assurance/quality control (QA/QC) for the FWS. Analytical methods for sediment and fish samples are described in the following paragraphs.

Ten-gram tissue samples were thoroughly mixed with anhydrous sodium sulfate and soxhlet extracted with hexane for seven hours. The extract was concentrated by rotary evaporation; transferred to a tared test tube, and further concentrated to dryness for lipid determination. The weighed lipid sample was dissolved in petroleum ether and extracted four times with acetonitrile saturated with petroleum ether. Residues were partitioned into petroleum ether which was washed, concentrated, and transferred to a

glass chromatographic column containing 20 grams of Florisil. The column was eluted with 200 ml 6% dimethyl ether/94% petroleum ether (Fraction I) followed by 200 ml 15% dimethyl ether/85% petroleum ether (Fraction II). Fraction II was concentrated to appropriate volume for quantification of residues by packed column electron capture gas chromatography. Fraction I was concentrated and transferred to a silicic acid chromatographic column for additional cleanup required for separation of polychlorinated biphenyls (PCBs) from other organochlorines. Three fractions were eluted from the silicic acid column. Each fraction was concentrated to appropriate volume for quantification of residues by packed or megabore column, electron capture gas chromatography. PCBs were found in Fraction II.

Twenty five-gram soil or sediment samples were extracted with acetone followed by hexane, by allowing to soak one hour in each with intermittent shaking. The combined extracts were centrifuged and decanted into a separatory funnel containing sufficient water to facilitate partitioning of 2 residues into hexane portion. The hexane was washed twice with water and concentrated to appropriate volume for transfer to a 1.6 gram Florisil mini-column topped with 1.6 grams sodium sulfate. Residues were eluted from the column in two elution fractions. Fraction I consists of 12 milliliters hexane followed by 12 milliliters of 1% methanol in hexane. If additional cleanup was required to separate PCBs from other organochlorines in Fraction I, further chromatography on a silicic acid column was performed as previously described. Quantification of residue in the two Florisil fractions and three silicic acid fractions was by packed or megabore column, electron capture gas chromatography.

#### Statistical Methods

Since one of the three fish from each station had been separated into fillet and carcass portions for analysis, a weighted mean value for whole body burden was derived to allow comparison of contaminant concentrations at each station. The weighted mean value for whole body burden was calculated using the following equation:

Whole body burden (ppm) = <u>Carcass wt. (ppm dry wt.) + Fillet wt. (ppm dry wt.)</u>

Total wt. (ppm)

Concentrations were converted to dry weight. Due to the relatively small sample sizes for each location (n = 3), nonparametric statistical tests were used to differentiate between and among median contaminant concentrations. Correlation coefficients (Pearson and Spearman rank-order correlation coefficients) were computed to determine if the samples had been biased by selecting the largest fish for separation into fillet and carcass portions. The Wilcoxon's signed-rank test was used to determine if significant differences existed between whole fish samples (n = 10) and calculated weighted mean samples (n = 5). A Kruskal-Wallis test was used to evaluate the differences occurring among all stations (Sokol and Rohlf 1981). Statistical significance was assumed at  $P \le 0.05$ . All data analyses were performed using the PC version of SAS (SAS Institute, Inc. 1985).

Table 2. Chlordane residue levels (ppm wet weight concentration) found in brown bullheads collected from the non-tidal portion of the Patuxent River.

Station P-1: 0.25 mile downstream from Route 198 bridge in Laurel

	1	2			3
Residue	whole fish	whole fish	carcass1 +	<u>fillet</u> <sup>1</sup> =	(wholefish)1
heptachlor epoxide trans-chlordane t-nonachlor cis-chlordane cis-nonachlor	ND ND 0.01 0.01 0.01 ND	0.03	ND ND 0.06 0.06 0.07 ND	ND 0.03 0.02 0.03 ND	0.09 0.08 0.10
total chlordane	0.03 79.8 2.23	76.0	.019 77.6 4.02	0.08 0.08 77.0 1.95	0.27

Station P-PWRC: Duvall Bridge, Patuxent Wildlife Research Center, Laurel

	1	2			3
Residue	whole fish1	whole fish1	carcass1 +	<u>fillet</u> =	(wholefish)
oxychlordane	ND ND	ND	ND	ND	
Heptachlor epoxide	ND	ND	ND .	ND	
Trans-chlordane	0.01	0.03	0.07	0.01	0.08
t-nonachlor	0.01	0.02	0.02	0.01	0.03
cis-chlordane	0.01	0.03	0.03	0.02	0.05
cis-nonachlor	ND	ND	ND	ND	
Total chlordane	0.03	0.08	0.12	0.04	0.16
FDA-based <sup>2</sup>	T-9 CT-	7,72	3.36	0.02	
% moisture	74.8	75.0	77.2	78.0	
% lipid	4.23	5.16	2.10	7.83	

Table 2. concluded

Station LP-3: 0.5 mile upstream of Route 3 bridge on Little Patuxent River

	level for chlo	2			3
Residue	whole fish1	whole fish1	carcass1 +	<u>fillet</u> 1 =	( <u>wholefish</u> ) <sup>1</sup>
oxychlordane heptachlor epoxide	ND ND	ND 0.02	ND ND	ND ND	
trans-chlordane	ND	0.02	0.01	0.01	0.02
t-nonachlor	ND	0.01	0.01	0.01	0.02
cis-chlordane	ND	0.02	0.01	0.01	0.02
cis-nonachlor	ND	ND	ND	ND	Data
Total chlordane	cons, with lave tations have to	0.07	0.03	0.03	0.06
FDA-based <sup>2</sup>				0.00	
% moisture	78.2	74.8	78.8	78.4	
% lipid	0.753	5.54	2.02	1.96	

Station LP-3: 0.5 mile downstream of Route 424 bridge on Little Patuxent River near Ft. Meade entrance

D/2		1	1	6/22 - 1	(4-1-61-1)
Residue	whole fish1	whole fish1	carcass1 +	<u>fillet</u> <sup>1</sup> =	(wholefish) 1
oxychlordane	ND	ND	ND	ND	
heptachlor epoxide	ND	0.01	ND	ND	
trans-chlordane	0.02	0.01	0.09	0.01	0.10
t-nonachlor	0.02	0.01	0.05	0.02	0.07
cis-chlordane	0.03	0.01	0.04	0.02	0.06
cis-nonachlor	ND	ND	ND	ND	swind .
total chlordane	0.07	0.04	0.18	0.05	0.23
FDA-based <sup>1</sup>				0.04	
% moisture	74.6	72.6	75.6	75.8	
% lipid	7.01	4.43	4.68	3.28	
					Dioritio

<sup>1 -</sup> ppm. Intersections and a total aspectation betogen at 10 mained (byes

Note: Sediment samples (composite of 2 grabs) were taken at all stations and none of the chlordane isomers were detected at any of the six stations.

<sup>2 -</sup> Levels of individual components must be quantitated at 0.02 ppm or above.

<sup>\* -</sup> These two samples were white catfish.

<sup>) -</sup> Summation of carcass and fillet. These whole fish concentrations were not not chemically analyzed. ND - Not detected.

bioaccumulate, logic dictates that residue levels in fish would continue to increase for several years following these bans, which seems to be the case on the Patuxent River based on available data.

20

In 1988, the FWS collected two brown bullheads at P-1, the station farthest upstream, which had whole body burdens of 0.03 ppm (wet weight) and 0.09 ppm (wet weight). These levels were lower than those found by MDE in both 1977-1978 and 1984. They are also lower than the 0.1 ppm limit set by the NAS-NAE for chlordane levels in whole fish, which indicates that these fish were within an allowable range of chlordane residue concentration. 1977 and 1978, two black crappie (Pomoxis nigromaculatus) specimens, collected from station PXT0809 (just upstream of P-1) by MDE had chlordane concentrations of 0.19 ppm and 0.02 ppm, respectively (Harmon 1980). In 1980, the State collected a spottail shiner (Notropis hudsonius) and a fallfish at station PXT0808 (also upstream of P-1), both with chlordane levels of 0.09 ppm (Allison and Butler 1982). At this same location in 1981, a pumpkinseed (Lepomis gibbosus) with a level of 0.01 ppm was collected, and in 1982 a white sucker with a level of 0.069 ppm was taken (Butler and Allison 1984). All but one of these fish were within the allowable chlordane concentration range set by NAS-NAE. The highest residue levels in fish at this site were found in 1984 when MDE collected specimens with the following levels: (1) white sucker - 0.36 ppm; and (2) fallfish - 0.35 ppm (Murphy 1986). A brown bullhead fillet collected by FWS from sample site P-1 and submitted for analysis in 1988 had a chlordane residue level of 0.08 ppm. This is comparable to levels found in the two whole fish samples collected from this station (0.03 ppm and 0.09 ppm, respectively). MDE has not submitted any fish fillets from this area for analysis.

Data collected from the Patuxent River near the Patuxent Wildlife Research Center between 1981 and 1988 further substantiate the idea that there is a temporal variation pattern of chlordane levels in non-tidal areas. Both MDE and FWS have collected fish in this vicinity (Stations PXT0728 and P-WRC, respectively). In 1988, the FWS collected two brown bullheads at this station with chlordane residue levels of 0.03 ppm and 0.08 ppm. The chlordane levels in these two specimens were lower than those in the fish collected by MDE in 1981 and 1983. In 1981, MDE collected a pumpkinseed and a white sucker with chlordane whole body burdens of 0.219 ppm and 0.319 ppm, respectively (Butler and Allison 1984). In 1983, a white sucker was collected with a 0.768 ppm level (Murphy 1986). Chlordane levels at this station appear to have peaked in 1983.

Station P-50 (MDE station PXT0603) is a site having some of the most extensive sampling data. Comparison of these data also reveals an increase in chlordane levels found through 1983, followed by a decrease, lending credence to the trend of temporal variation in chlordane levels previously mentioned. Two brown bullheads collected at station P-50 by FWS in 1988 both had whole body burdens of chlordane of 0.04 ppm. These are some of the lowest residue levels found in any species at this site since 1982. In 1978-79, MDE collected a bluegill, white sucker, and carp (Cyprinus carpio) with chlordane whole body burdens of 0.16 ppm, 0.48 ppm, and 0.26 ppm, respectively. In 1981, MDE reported residue levels ranging from 0.031 ppm

Patuxent River in the future, however periodic fish analysis will be required to determine this.

22

Analysis of the geographic distribution of brown bullhead chlordane residues in the non-tidal portion of the Patuxent River indicated a decreasing trend progressing from upstream to downstream stations. This seems to be a logical pattern when related to the degree of development adjacent to the Patuxent along this stretch. The upstream portion near Laurel, Maryland is more urbanized while the lower sections south to Davidsonville are more buffered, with potential sources of chlordane further from the mainstem of the Patuxent. For example, large areas of the Patuxent River (PWRC and Patuxent River State Park) support large forested buffer strips protecting the river from potential non-point run-off (i.e. stormwater) from surrounding suburbs such as Laurel, Bowie, and Davidsonville, Maryland.

#### CONCLUSIONS AND RECOMMENDATIONS

Comparison of MDE data and FWS data for residue levels in the same species were possible at two stations (P-50 and LP-3), since MDE only caught brown bullheads at PXT0603 and PXT0630. These results reveal that residue levels in brown bullheads collected in 1988 were lower than those in the same species collected from the same areas several years earlier. The remaining data allow only inter-species comparisons, but they do indicate a general trend of lower chlordane residue levels in the 1988 specimens.

Extreme variability in the 1988 results indicate that the geographic distribution of chlordane does not represent a significant direct relationship. The fact that the geographic pattern indicated by this study proved not to be significant might be explained by various factors. Collecting more specimens at each station would undoubtedly provide more reliable results, however, budget constraints restricted the number of specimens that could be analyzed. This information may reflect that sample locations were too close to show a significant trend or that the same fish population was being sampled due to the proximity of sample locations.

While this information proves beneficial as baseline data for future monitoring of chlordane in the Patuxent River, a larger scale (more samples per station, more stations, larger section of the river) study would need to be conducted to fully ascertain geographic distribution of chlordane in the Patuxent. Currently, it would appear that no health risk associated with chlordane bioaccumulation in fish exists based on this study's results.

- 24
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Appendix A: Lengths and weights of Patuxent River samples submitted for chlordane analysis

Sample ID#	Species	Total Length (mm)	Total Weight (g)	
P1BA	brown bullhead	253	190	
PIBB	brown bullhead	220	190	9
P1BC*	brown bullhead	242	220	
PIS	sediment		25	
PWRCBA	brown bullhead	271	240	
PWRCBB	brown bullhead	273	302	
PWRCBC*	brown bullhead	220	132	
PWRCS	sediment		25	
P50BA	brown bullhead	314	297	
P50BB	brown bullhead	281	297	
P50BC*	brown bullhead	289	347	
P50S	sediment		25	
P214BA	brown bullhead	315	363	
P214BB	brown bullhead	292	388	
P214BC*	brown bullhead	345	585	
P214S	sediment		25	

Appendix A. concluded

Sample ID#	Species	Total Length (mm)	Total Weight (g)	
LP3BA	brown bullhead	211	179	
LP3BB	brown bullhead	296	339	
LP3BC*	brown bullhead	264	260	
LP3S	sediment		25	
LPFMBA	brown bullhead	314	419	
LPFMWA	white catfish	315	425	
LPFMWB*	white catfish	330	488	
LPFMS	sediment		25	

<sup>\*</sup> Separated for analysis of fillet and carcass portions.

Appendix B. Analytical results of chemical analyses of fish and sediment samples from the Patuxent River

MISSISSIPPI STATE UNIVERSITY MISSISSIPPI STATE CHEMICAL LABORATORY

BOX CR MISSISSIPPI STATE, MS 39762 REPORT FORM USDI/FWS

DATE RECEIVED 10/24/88

Page 1

**ORGANOCHLORINES** 

PARTS PER MILLION AS RECEIVED (WET WT) P WRCBB P 1BA P 1BB P 1S FWS # P 1BCC P 1BCF P WRCBA 758291 758292 LAB # 758286 758287 758288 758289 758290 B. B. Brown Brown B. B. Brown Brown MATRIX Fillet Sediment Bullhead Bullhead Bullhead Bullhead Carcass COMPOUND HCB ND\* ND ND ND ND ND ND α−BHC ND ND ND ND ND ND ND ND ND r-BHC ND ND ND ND ND g -BHC ND ND ND ND ND ND ND &-BHC ND ND ND ND ND ND ND Oxychlordane ND Hept. Epox. ND ND ND r-Chlordane 0.03 0.01 0.02 0.06 ND 0.01 0.03 t-Nonach Ior 0.01 0.03 0.02 ND 0.01 0.02 0.06 ND ND ND ND Toxaphene ND ND ND 0.05 0.06 0.33 PCB's (total) 0.40 0.23 ND 0.12 o, p'-DDE ND ND ND ND ND ND ND α-Chlordane 0.01 0.03 0.07 0.03 ND 0.01 0.03 p, p'-DDE 0.03 0.01 0.04 0.01 0.02 0.03 ND 0.01 0.01 Dieldrin ND ND 0.01 ND ND o, p'-DDD ND ND ND ND ND ND ND Endrin ND cis-nonachlor ND ND ND o, p'-DDT ND ND ND ND ND ND ND p, p'-DDD 0.06 0.01 0.03 0.01 ND 0.01 0.02 p, p'-DDT 0.01 0.01 0.01 0.01 ND 0.01 0.01 Mirex ND ND ND ND ND ND ND OTHER: WEIGHT (g) 189 194 177 36.9 179 234 298 MOISTURE (%) 79.8 76.0 77.6 77.0 74.8 75.0 32.6 LIPID (%) 2.23 2.68 4.02 1.95 4.23 5.16

Lower Level of Detection = 0.01 ppm for Tissue, Soil, Etc. 0.05 for Toxaphene and PCBs. For Water, LLD= 0.005 ppm for OCs, Tox , PCBs

\*\*Spike = ppm for

# = Confirmed by GC/Mass Spectrometry

\*ND = None Detected

\*\*\*NS = Not Spiked

SAMPLE TYPE: Brown

BATCH NO. 88-5-043 ORDER NO. 85800-88-

Catfish, and CAT NO. 5587

Bullhead, White Catfish, and Sediment

30035

Signature

MISSISSIPPI STATE CHEMICAL LABORATORY
BOX CR
MISSISSIPPI STATE, MS 39762
REPORT FORM
USDI/FWS

SAMPLE TYPE: Bro Bullhead, White Catfish, and Sec CAT NO. 5587 BATCH NO. 88-5-0	own liment	MISSISS	Page				
ORDER NO. 85800- 30035	-88-	Reser	RGANOCHLOR			ATE RECEIV	ED 10/24/8
	PA	RTS PER M	ILLION AS	RECEIVED (	WET WT)		
FWS #	P WRCBCC	P WRCBF	P WRCS	P 50BA	P 50BB	P 50BCC	P 50BCF
LAB #	758293	758294	758295	758296	758297	758298	758299
MATRIX	B. B. Carcass			Brown Bullhead	B. B. carcass	B. B. Fillet	
COMPOUND							
нсв	ND*	ND	ND	ND	ND	ND	ND
α-ВНС	ND	ND	ND	ND	ND	ND	ND
r-BHC	ND	ND	ND	ND	ND	ND	ND
β -BHC	ND	ND	ND	ND	ND	ND	ND
§-BHC	ND	ND	ND	ND	ND	ND	ND
Oxychlordane	ND	ND	ND	ND .	ND	ND	ND
Hept. Epox.	ND	ND	ND	ND	ND	ND	ND
r-Chlordane	0.07	0.01	ND	0.01	0.01	0.02	0.01
t-Nonachlor	0.02	0.01	ND	0.01	0.02	0.02	0.01
Toxaphene	ND	ND	ND	ND	ND	ND	ND
PCB's (total)	0.30	0.19	ND	0.37	0.33	0.29	0.17
o, p'-DDE	ND	ND	ND	ND	ND	ND	ND
α-Chlordane	0.03	0.02	ND	0.02	0.01	0.02	0.02
p, p'-DDE	0.02	0.01	ND	0.04	0.02	0.02	0.02
Dieldrin	0.01	ND	ND	ND	ND	0.01	0.01
o, p'-DDD	ND	ND	ND	ND	ND	ND	ND
Endrin	ND	ND	ND	ND	ND	ND	ND
cis-nonachlor	ND	ND	ND	ND	ND	ND	ND
o, p'-DDT	ND	ND	ND	ND	ND	ND	ND
p, p'-DDD	0.01	0.01	ND	0.01	0.01	0.01	0.01
p, p'-DDT	0.01	0.01	ND	0.01	0.01	0.01	0.01
Mirex	ND	ND	ND	ND	ND	ND	ND
OTHER:				_			
WEIGHT (g)	101	21.0	173	335	291	281	52.8
MOISTURE (%)	77.2	78.0	36.8	75.4	79.8	75.4	78.2
LIPID (%)	2.10	7.83	-	5.23	2.27	4.04	2.90

Lower Level of Detection = 0.01 ppm for Tissue, Soil, Etc. 0.05 for Toxaphene and PCBs.

For Water, LLD= 0.005 ppm for OCs, Tox, PCBs

\*\*Spike = ppm for

# = Confirmed by GC/Mass Spectrometry

\*ND = None Detected

\*\*\*NS = Not Spiked

21

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21

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MISSISSIPPI STATE CHEMICAL LABORATORY
BOX CR
MISSISSIPPI STATE, MS 39762
REPORT FORM
USDI/FWS

ORGANOCHLORINES

DATE RECEIVED 10/24/88

Page 3

DADTO BED WILLIAM AC BECEIVED CHET WITH

FWS #	P 50S	P 214BA	P 214BB	P 214BB	P 214BCC	P 214BCF	P 214S
LAB *	758300	758301	758302A	758302B	758303	758304	758305
MATRIX	Sediment	Brown Bullhead	Brown Bullhead	Duplicate B.Bullhea	B. B. Carcass	B. B. Fillet	Sediment
COMPOUND							
НСВ	ND*	ND .	ND	ND	ND	ND	ND
α-BHC	ND	ND	ND	ND	ND	ND	ND
r-BHC	ND	ND	ND	ND	ND	ND	ND
β-BHC	ND	ND	ND	ND	ND	ND	ND
δ-BHC	ND	ND	ND	ND	ND	ND	ND
Oxychlordane	ND	ND	ND	ND .	ND	ND	ND
Hept. Epox.	ND	ND	ND	ND	ND	0.01	ND
r-Chlordane	ND	0.01	0.01	0.01	0.04	0.02	ND
t-Nonachlor	ND	0.01	0.01	0.01	0.09	0.07	ND
Toxaphene	ND	ND	ND	ND	ND	ND	ND
PCB's (total)	ND	0.15	0.18	0.19	1.4#	1.3#	ND
o, p'-DDE	ND	ND	ND	ND	ND	ND	ND
α-Chlordane	ND	0.01	0.01	0.01	0.07	0.05	ND
p, p'-DDE	ND	0.01	0.02	0.02	0.19#	0.15#	ND
Dieldrin	ND	ND	0.01	0.01	0.02	0.02	ND
o, p'-DDD	ND	ND	- ND	ND	ND	ND	ND
Endrin	ND	ND	ND	ND	ND	ND	ND
cis-nonachlor	ND	ND	ND	ND	ND	ND	ND
o, p'-DDT	ND	ND	ND	ND	ND	ND	ND
p, p'-DDD	ND	ND	0.01	0.01	ND	0.04	ND
p, p'-DDT	ND	ND	0.01	0.01	ND	ND	ND
Mirex	ND	ND	ND	ND	ND	ND	ND
OTHER:		1					
WEIGHT (g)	143	489	383	383	509	68.0	165
MOISTURE (%)	51.2	75.8	72.0	72.2	73.2	76.2	49.0
LIPID (%)		3.36	7.23	8.13	6.17	5.53	-

Lower Level of Detection = 0.01 ppm for Tissue, Soil, Etc. 0.05 for Toxaphene and PCBs.

For Water, LLD= 0.005 ppm for OCs, Tox, PCBs

\*\*Spike = ppm for

# = Confirmed by GC/Mass Spectrometry

\*ND = None Detected

\*\*\*NS = Not Spiked

22

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22

SAMPLE TYPE: Brown Bullhead, White Catfish, and Sediment CAT NO. 5587 BATCH NO. 88-5-043 ORDER NO. 85800-88-

30035

SAMPLE TYPE: Brown Bullhead, White Catfish, and Sediment CAT NO. 5587 BATCH NO. 88-5-043 ORDER NO. 85800-88-

30035

# MISSISSIPPI STATE UNIVERSITY MISSISSIPPI STATE CHEMICAL LABORATORY BOX CR MISSISSIPPI STATE, MS 39762 REPORT FORM USDI/FWS

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DATE RECEIVED 10/24/88

Page 4

PARTS PER MILLION AS RECEIVED (WET WT)

FWS #	LP 3BA	LP 3BB	LP 3BCC	LPB3CF	LP 3S	LP FMBA	LP FMWA
LAB *	758306	758307	758308	758309	758310	758311	758312
MATRIX	Brown Bullhead	Brown Bullhead	B. B. Carcass	B. B. Fillet	Sediment	Brown Bullhead	White Catfish
COMPOUND							
нсв	ND*	ND .	ND	ND	ND	ND	ND
α-BHC	ND	ND	ND	ND	ND	ND	ND
r-BHC	ND	ND	ND	ND	ND	ND	ND
<sub>в</sub> –ВНС	ND	ND	ND	ND	ND	ND	ND
6-BHC	ND	ND	ND	ND	ND	ND	ND
Oxychlordane	ND	ND	ND	ND .	ND	ND	ND
Hept. Epox.	ND	0.02	ND	ND	ND	ND	0.01
r-Chlordane	ND	0.02	0.01	0.01	ND	0.02	0.01
t-Nonach For	ND	0.01	0.01	0.01	ND	0.02	0.01
Toxaphene	ND	ND	ND	ND	ND	ND	ND
PCB's (total)	0.12	0.19	0.21	0.23	ND	0.32	0.10
o, p'-DDE	ND	ND	ND	ND	ND	ND	ND
α-Chlordane	ND	0.02	0.01	0.01	ND	0.03	0.01
p, p'-DDE	0.01	0.03	0.01	0.02	ND	0.03	0.02
Dieldrin	ND	0.01	ND	ND	ND	ND	ND
o, p'-DDD	ND	ND	- ND	ND	ND	ND	ND
Endrin	ND	ND	ND	ND	ND	ND	ND
cis-nonachior	ND	ND	ND	ND	ND .	ND	ND
o, p'-DDT	ND	ND	ND	ND	ND	ND	ND
p, p'-DDD	ND	0.01	ND	ND ·	ND	0.01	0.02
p, p'-DDT	ND	ND	ND	ND	ND	ND	ND
Mirex	ND	ND	ND	ND	ND	ND	ND
OTHER:							
WEIGHT (g)	177	333	216	38.5	173	379	425
MOISTURE (%)	78.2	74.8	78.8	78.4	35.2	74.6	72.6
LIPID (%)	0.753	5.54	2.02	1.96	_	7.01	4.43

Lower Level of Detection = 0.01 ppm for Tissue, Soil, Etc. 0.05 for Toxaphene and PCBs.

For Water, LLD= 0.005 ppm for OCs, Tox, PCBs

\*\*Spike = ppm for

# = Confirmed by GC/Mass Spectrometry

\*ND = None Detected

\*\*\*NS = Not Spiked

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MISSISSIPPI STATE UNIVERSITY
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BOX CR
MISSISSIPPI STATE, MS 39762
REPORT FORM

USDI/FWS

Page 5

ORGANOCHLORINES

DATE RECEIVED 10/24/88

FWS #	LP FMWBC	LP FMWCF	LP FMS	Blank	Matrix Blank	Spike**	% Recover:
LAB #	758313	758314	758315	758316	for	758317	
MATRIX	W.Catfish Carcass	W.Catfish Fillet	Sediment	Reagent	Fish	Fish	
COMPOUND							
нсв	ND*	ND	ND	ND	ND	0.060	60
α-ВНС	ND	ND	ND	ND	ND	NS***	
-BHC	ND	ND	ND	ND	ND	0.093	93
<sub>B</sub> -BHC	ND	ND	ND	ND	ND	0.092	92
6-BHC	ND	ND	ND	ND	ND	NS	
Oxychlordane	ND	ND	ND	ND .	ND	0.085	85
Hept. Epox.	ND	ND	ND	ND	ND	0.090	90
-Chlordane	0.09	0.01	ND	ND	ND	NS	
t-Nonach I or	0.05	0.02	ND	ND	ND	0.086	86
Toxaphene	4.7*	ND	ND	ND	ND	NS	
PCB's (total)	ND	0.40	ND	ND	ND	NS	
o, p'-DDE	ND	ND	ND	ND	ND	0.093	93
α-Chlordane	0.04	0.02	ND	ND	ND	0.093	93
p, p'-DDE	10.#	0.05	ND	ND	0.01	0.10	100
Dieldrin	0.03	0.01	ND	ND	ND	0.090	90
o, p'-DDD	ND	ND	ND	ND	ND	NS	
Endrin	ND	ND	ND	ND	ND	0.093	93
cis-nonachior	ND	ND	ND	ND	ND	0.098	98
o, p'-DDT	ND	ND	ND	ND	ND	0.10	100
p, p'-DDD	3.2#	0.03	ND	ND	ND	0.092	92
p, p'-DDT	0.77*	ND	ND	ND	ND	0.097	97
Mirex	ND	ND	ND	ND	ND	0.090	90
OTHER:							
WEIGHT (g)	422	54.0	167	-	-		-
MOISTURE (%)	75.6	75.8	37.0	-	77.6	79.0	-
LIPID (%)	4.68	3.28	-	-	1.46	1.41	-

Lower Level of Detection = 0.01 ppm for Tissue, Soil, Etc. 0.05 for Toxaphene and PCBs.

For Water, LLD= 0.005 ppm for OCs, Tox, PCBs

\*\*Spike = 0.10 ppm for Fish

# = Confirmed by GC/Mass Spectrometry

\*ND = None Detected

\*\*\*NS = Not Spiked

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24

SAMPLE TYPE: Brown Bullhead, White Catfish, and Sediment CAT NO. 5587 BATCH NO. 88-5-043 ORDER NO. 85800-88-

30035

MISSISSIPPI STATE CHEMICAL LABORATORY
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REPORT FORM
USDI/FWS

SAMPLE TYPE: Bro Bullhead, White Catfish, and Sed CAT NO. 5587	Iment	MISSISSIPPI STATE, MS 39762 Page REPORT FORM USDI/FWS					
BATCH NO. 88-5-0 ORDER NO. 85800- 30035	88-		RGANOCHLOR			ATE RECEIV	ED 10/24/
	P,	ARTS PER M	T	RECEIVED (			T
FWS #	Blank	Blank	Matrix Blank	Spike**	% Recovery	Blank	Matrix Blank
LAB #	758318	758319	for	758320		758321	for
MATRIX	Reagent	Reagnet	Fish	Fish		Reagent	Sediment
COMPOUND							
НСВ	ND*	ND .	ND	0.066	66	ND	ND
α-ВНС	ND	ND	ND	NS***		ND	ND
r-BHC	ND	ND	ND	0.099	99	ND	ND
ρ -BHC	ND	ND	ND	0.097	97	ND	ND
6-BHC	ND	ND	ND	NS		ND	ND
Oxychlordane	ND	ND	ND	0.091	91	ND	ND
Hept. Epox.	ND	ND	ND	0.098	98	ND	ND
r-Chlordane	ND	ND	ND	NS		ND	ND
t-Nonach Ior	ND	ND	ND	0.091	91	ND	ND
Toxaphene	ND	ND	ND	NS		ND	ND
PCB's (total)	ND	ND	ND	NS		ND	ND
o, p'-DDE	ND	ND	ND	0.099	99	ND	ND
α-Chlordane	ND	ND	ND	0.099	99	ND	ND
p, p'-DDE	ND	ND	0.01	0.10	100	ND	ND
Dieldrin	ND	ND	ND	0.096	96	ND	ND
o, p'-DDD	ND	ND	- ND	NS		ND	ND
Endrin	ND	ND	ND	0.095	95	ND	ND
cis-nonachlor	ND	ND	ND	0.10	100	ND	ND
o, p'-DDT	ND	ND	ND	0.11	110	ND	ND
p, p'-DDD	ND	ND	ND	0.10	100	ND	ND
p, p'-DDT	ND	ND	ND	0.098	98	ND	ND
Mirex	ND	ND	ND	0.086	86	ND	0.01
OTHER:							
WEIGHT (g)	-	-	-	-	-	-	-
MOISTURE (%)	-	-	76.2	74.8	-	-	50.0
LIPID (%)	_	-	3.12	3.19	-	-	-

Lower Level of Detection = 0.01 ppm for Tissue, Soil, Etc. 0.05 for Toxaphene and PCBs.

For Water, LLD= 0.005 ppm for OCs, Tox, PCBs

\*\*Spike = 0.10 ppm for Fish

# = Confirmed by GC/Mass Spectrometry

\*ND = None Detected

\*\*\*NS = Not Spiked

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25

# MISSISSIPPI STATE UNIVERSITY MISSISSIPPI STATE CHEMICAL LABORATORY BOX CR MISSISSIPPI STATE, MS 39762 REPORT FORM USDI/FWS

SAMPLE TYPE: Bro Bullhead, White Catfish, and Sec CAT NO. 5587 BATCH NO. 88-5-0 ORDER NO. 85800- 30035	diment	SSISSIPPI MISSISS	STATE CHEMI BOX CR SIPPI STATE, REPORT FOR USDI/FWS	ICAL LABORA , MS 39762 RM		DATE RECEIV	Page 7
00000			MILLION AS F	RECEIVED (W	ET WT)		
FWS #	Spike**	% Recovery					
LAB #	758322						
MATRIX	Sediment						
COMPOUND							
нсв	0.030	75	•				T
α-BHC	NS***						
r-BHC	0.032	80					
β -BHC	0.040	100	1				
6-BHC	NS						
Oxychlordane	0.035	88					
Hept. Epox.	0.036	90					
r-Chlordane	NS						
t-Nonach Ior	0.043	108					
Toxaphene	NS	NS					
PCB's (total)	NS	NS					
o, p'-DDE	0.041	103					
α-Chlordane	0.037	93					
p, p'-DDE	0.042	105					
Dieldrin	0.038	95					
o, p'-DDD	NS						
Endrin	0.040	100					
cis-nonachlor	0.044	110					
o, p'-DDT	0.035	88					
p, p'-DDD	0.041	103					
p, p'-DDT	0.036	90					
Mirex	0.042	105					
OTHER:							
WEIGHT (g)	-	-					
MOISTURE (%)	50.0	-					
LIPID (%)	-	-					

Lower Level of Detection = 0.01 ppm for Tissue, Soil, Etc. 0.05 for Toxaphene and PCBs.

For Water, LLD= 0.005 ppm for OCs, Tox, PCBs

\*\*Spike = 0.040 ppm for Sediment

# = Confirmed by GC/Mass Spectrometry

\*ND = None Detected

\*\*\*NS = Not Spiked

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26