

**Report to FFOU on Polychlorinated Biphenyl (PCB)
Residues in Fish from the Bayou Creek System**

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FINAL REPORT

Fish were collected from Little Bayou Creek on September 29-30, 1997. Fillet samples from 5 sunfish were analyzed for Aroclors 1248, 1254, and 1260 for each of 4 stream stations. PCB assays for water and sediments from the Bayou system were given previously in the December report (Birge and Price, 1997).

METHODS

Fish collection

Fish were collected by use of back-pack shockers. Collections were conducted by UK; FFOU; and Fish and Wildlife personnel. Fish that did not meet our requirements were returned to the stream. Collected fish were wrapped in aluminum foil, tagged, bagged, and placed on ice (4 °C) for transport to the laboratory. Fish species were identified and stored in the freezer (-15 °C) until extraction.

Tissue extraction and clean-up.

Fish were measured for length and whole body weight, scales were removed, and fillets were taken with solvent-cleaned surgical instruments. The fillets were then weighed and macerated as described below. Otoliths (sagittae) were removed from each specimen for age determinations (Boxrucker 1986).

PCBs in fish tissues were extracted and analyzed using standard U.S. EPA methods (Watts, 1980; U.S. EPA, 1996; Erickson, 1997). The muscle fillet samples were ground with 10g anhydrous sodium sulfate and the powder extracted with petroleum ether in a Soxhlet apparatus for 5-h. The extracts were concentrated to near dryness in a Roto-evaporator (Buchi Model RE121). Reconstituted samples (5.0 mL in iso-octane) were then cleaned of interferences as described below and then analyzed by gas chromatography. Lipid and pesticide clean-up was performed by eluting a 2.0 mL sample through a micro-column of 2.0 g activated 100-200 mesh Florisil® (100 °C/24 h) with 10.0 mL 6% ethyl ether in petroleum ether and evaporated to 2.0 mL (Erickson, 1997; U.S. EPA, 1996, SW-846 Method 3620B, Florisil cleanup). Elemental sulfur was then removed by shaking 2-propanol (2 mL) and tetrabutylammonium sulfite (2 mL), adding ultra-pure water (8 mL) and reshaking. The organic extract was removed and mixed with 2.0-mL concentrated sulfuric acid (Jensen *et al.*, 1977; U.S. EPA, 1996, SW-846 Method 3660B, sulfur cleanup). A 4 µL sub-sample was then analyzed by gas chromatography.

Analysis by Gas Chromatography

Samples were analyzed for Aroclors 1248, 1254, and 1260 according to SW-846 Method 8082, polychlorinated biphenyls by gas chromatography (U.S. EPA, 1996). Analysis was performed using a Hewlett-Packard (HP) Model 5890A gas chromatograph equipped with an electron capture detector and an HP Model

7673A Automatic Sampler. Samples were analyzed using a 60m X 0.53mm ID SPB-5 (0.5 μ m film) fused silica megabore column (Supelco, Inc.) with ultra-high purity helium and nitrogen as carrier and makeup gases, respectively. The temperature program was set at 160 °C (6 min)-10 °C/min-235 °C (0 min)-0.9 °C/min-260 °C (10 min); Injector temperature, 280 °C; Detector temperature, 300 °C. PCB peak heights were quantified using an HP Model 3396A integrator and multiple-peak linear regression analysis was performed with Lotus-123® software. Aroclor levels were calculated from heights of 6 to 9 peaks for Aroclors 1248 and 1260 and 4-6 peaks for Aroclor 1254. Five external standards were used for calibration curves and for every tenth sample either a solvent blank or a standard was analyzed. The Lotus program regresses data from PCB standards to the sample being analyzed. Each peak selected for each Aroclor class was statistically analyzed (*e.g.*, standard deviation; standard error; relative deviation).

Quality Assurance

All chain of custody and records are maintained in active files and are available for review by FFOU or the Cabinet for Natural Resources. Quality assurance for PCB assays included solvent blanks, procedure controls and spiked tissue recoveries (U.S. EPA, 1987).

RESULTS

PCB residues were detected in all fish analyzed. Except in the green sunfish, fillet concentrations generally were highest for Aroclor 1248, intermediate for Aroclor 1254 and lowest for Aroclor 1260 (Table 1, Figures 1-3). Among the three species of sunfish analyzed, PCB tissue concentrations were highest in the longear sunfish (Table 1, LS), followed in order by the bluegill (BG) and the green sunfish (GS). The latter is known to be more efficient than most fish in metabolizing pesticides and PCBs (Sanborn, J.R. *et al.*, 1975; Birge, *et al.*, 1992).

Total PCBs exceeded the action level of 2.0 mg/Kg (FDA, 1987) in one of five fish from station LB2 and a second specimen contained about 1.7 mg/Kg PCB. It should be noted that the largest specimens collected were less than two years old at the stream stations near PGDP effluents 011 and 010. The absence of older sunfish and sizable bottom feeders was attributed to low stream discharge. There was no appreciable flow at the upstream reference station (LB1) and fish acceptable for analysis were not present. Perhaps the next fish collection should be scheduled for late May or June 1998. This should provide a better assemblage of fish for PCB analysis.

Results obtained with fish analysis correlate with previous findings reported for water and sediment samples from Little Bayou Creek. As summarized in the December report (Birge and Price, 1997), PCBs were detected in the water column at stations LB2A, LB2 and LB3 in July 1997. Highest PCB sediment contamination also was observed at these monitoring sites. The frequency of action level fish was lower than reported in earlier years

(Birge *et al.*, 1992). However, this trend should be confirmed by the analysis of older and larger fish, as noted above.

QUALITY ASSURANCE

Quality assurance assays are summarized in Tables A1-A4. No contamination was observed for glassware (Table A1). Recoveries of PCBs in solvent were within 84-112% of that expected (Table A2). Tissue recoveries varied from 74 to 116% (Table A3). Duplicate assays were conducted with 4 fish, one from each station (Table A4). Assays A and B include PCB determinations of left and right fillets, respectively. Results were highly consistent.

REFERENCES

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Table 1. PCB Concentrations in Sunfish from Little Bayou Creek Collected September 29-30, 1997.

Sample Name	Length (mm)	Whole Body Wt. (g)	Age (Years)	Fillet Wt. (g)	mg fat /g tissue	Aroclor Conc. ($\mu\text{g/g}$)			
						1248	1254	1260	Total
LB2A093097PFBG1A	108	21.00	1+	3.726	2.68	0.229	0.116	0.062	0.407
LB2A093097PFBG2A	91	13.41	>1	2.431	4.11	<0.082	0.198	0.106	0.304
LB2A093097PFLS1A	104	24.63	>1	4.367	4.89	0.586	0.206	0.145	0.937
LB2A093097PFLS2A	88	13.28	>1	2.301	3.76	0.550	0.200	0.124	0.874
LB2A093097PFGS1A	101	16.85	>1	2.715	2.52	<0.074	0.225	0.103	0.328
LB2092997PFBG1A	101	20.61	>1	3.635	3.85	0.486	0.189	0.097	0.772
LB2092997PFLS1A	121	33.39	1+	4.860	2.90	0.980	0.351	0.359	1.691
LB2092997PFLS2A	112	28.89	1	4.200	2.46	1.624	0.400	0.431	2.456
LB2092997PFGS1A	121	26.03	1+	4.119	2.56	<0.049	0.191	0.334	0.525
LB2092997PFGS2A	100	17.30	>1	2.879	3.25	<0.069	0.120	0.056	0.176
LB3093097PFBG1A	97	17.15	>1	3.246	5.11	0.306	0.179	0.066	0.551
LB3093097PFBG2A	85	10.04	>1	1.488	4.64	<0.134	<0.134	<0.134	<0.134
LB3093097PFLS1A	115	29.27	1+	5.009	3.71	0.511	<0.040	0.085	0.596
LB3093097PFLS2A	110	26.76	1	5.032	4.19	0.377	0.138	0.075	0.590
LB3093097PFGS1A	135	36.84	2+	5.420	2.31	<0.037	0.119	0.092	0.211
LB4093097PFBG1A	101	17.25	2+	2.134	4.92	<0.094	<0.094	<0.094	<0.094
LB4093097PFLS1A	133	50.88	2+	6.832	3.62	<0.029	0.067	0.024	0.091
LB4093097PFGS1A	151	61.20	3+	10.076	2.82	0.108	0.066	0.024	0.197
LB4093097PFGS2A	139	51.21	2+	8.126	2.92	<0.025	0.062	0.032	0.095
LB4093097PFGS3A	134	35.79	2+	6.099	3.34	<0.033	0.129	0.055	0.184

Figure 1. PCB Concentrations in Longear Sunfish from Little Bayou Creek Collected September 29-30, 1997.

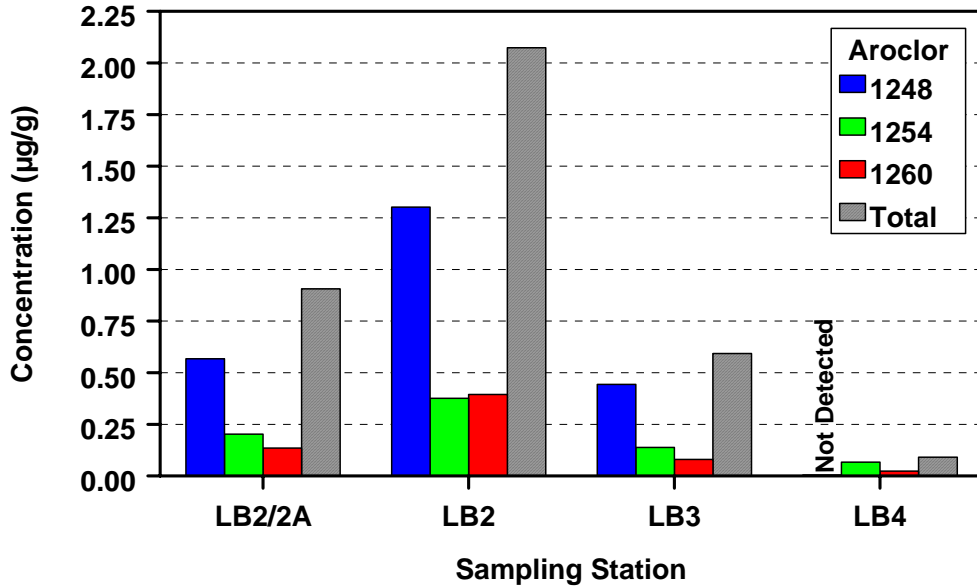


Figure 2. PCB Concentrations in Bluegill from Little Bayou Creek Collected September 29-30, 1997.

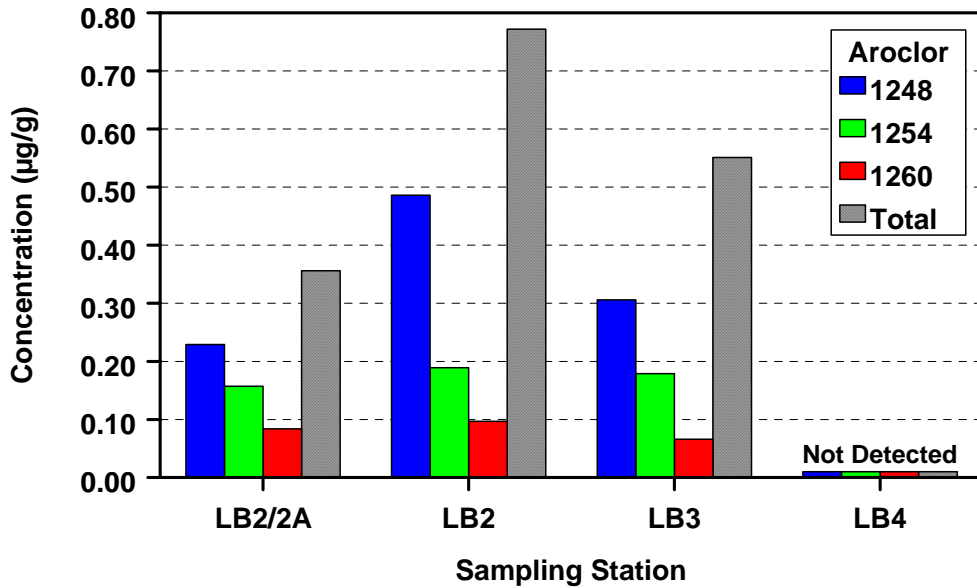


Figure 3. PCB Concentrations in Green Sunfish from Little Bayou Creek Collected September 29-30, 1997.

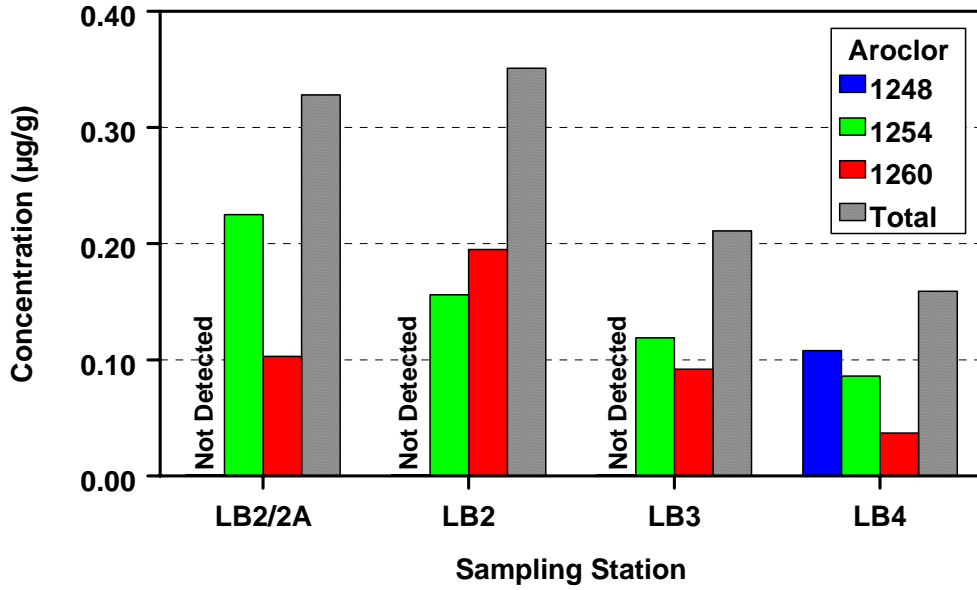


Figure 4. Total Aroclor Concentrations in Sunfish from Little Bayou Creek Collected September 29-30, 1997.

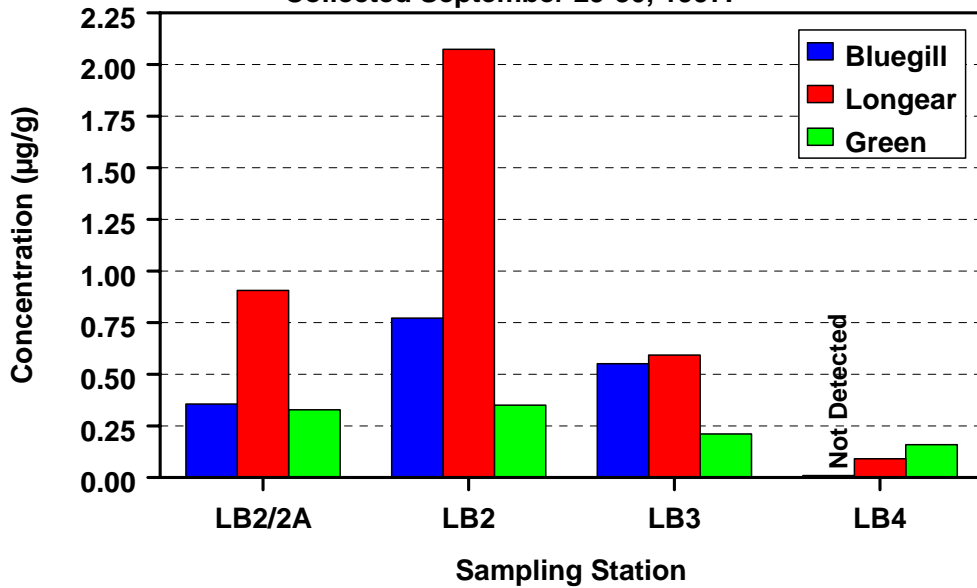


Table A1. PCB Concentrations in Controls for Fish from Little Bayou Creek Collected September 29-30, 1997.

Sample Name	Aroclor Conc. ($\mu\text{g}/\text{mL}$)		
	1248	1254	1260
CON120297PCON1F	<0.001	<0.001	<0.001
CON120297PCON2F	<0.001	<0.001	<0.001
CON120297PCON3F	<0.001	<0.001	<0.001
CON120297PCON2F	<0.001	<0.001	<0.001
CON120397PCON4F	<0.001	<0.001	<0.001
CON121297PCON5F	<0.001	<0.001	<0.001
CON121297PCON6F	<0.001	<0.001	<0.001
CON010598PCON1A	<0.001	<0.001	<0.001
CON010598PCON2A	<0.001	<0.001	<0.001

Table A2. PCB Concentrations in Procedure Recoveries ^A for Fish from Little Bayou Creek Collected September 29-30, 1997.

Sample Name	Aroclor	µg Spike	Aroclor Conc. (µg)		Percent Recovery
			Expected Conc.	Measured Conc.	
RECO120497PPRR1	1248	1.0	1.000	0.839	83.9
RECO120497PPRR2	1254	1.0	1.000	1.117	111.7
RECO010598PPRR1	1248	1.0	1.000	0.912	91.2
RECO010598PPRR2	1260	1.0	1.000	1.033	103.3

^A 300 mL of solvent were spiked with the respective Aroclor, extracted and analyzed as a regular sample.

Table A3. PCB Concentrations in Spiked Recoveries^A for Fish from Little Bayou Creek Collected September 29-30, 1997.

Sample Name	Aroclor	µg Spike	Aroclor Conc. (µg/mL)		Final Conc. (µg)	Percent Recovery
			Measured Conc.	Background Conc.		
LB4093097PSBG1B	1248	1.0	0.148	N.D. ^B	0.740	74.0
LB4093097PSGS3B	1254	1.0	0.283	0.050	1.162	116.2
LB4093097PSLS1B	1260	1.0	0.231	0.014	1.085	108.5

^A Fillet samples were spiked with the respective Aroclor, extracted, and analyzed.

^B N.D. no Aroclor detected.

Table A4. PCB Concentrations in Two Separate Fillets from Individual Sunfish from Little Bayou Creek Collected September 29-30, 1997.

Sample Name	Length (mm)	Whole Body Wt. (g)	Age (Years)	Fillet Wt. (g)	mg fat /g tissue	Aroclor Conc. (µg/g)			
						1248	1254	1260	Total
LB2A093097PFGS1A ^A	101	16.85	>1	2.715	2.52	<0.079	0.225	0.103	0.328
LB2A093097PFGS1B	101	16.85	>1	2.661	N.A. ^A	<0.075	0.196	0.085	0.281
LB2092997PFLS1A	121	33.39	1+	4.860	2.90	0.980	0.351	0.359	1.691
LB2092997PFLS1B	121	33.39	1+	4.219	N.A.	1.070	0.364	0.392	1.826
LB3093097PFGS1A	135	36.84	2+	5.420	2.31	<0.037	0.119	0.092	0.211
LB3093097PFGS1B	135	36.84	2+	5.586	N.A.	<0.036	0.130	0.092	0.222
LB4093097PFGS1A	151	61.20	3+	10.076	2.82	0.108	0.066	0.024	0.197
LB4093097PFGS1B	151	61.20	3+	8.598	N.A.	0.093	0.041	0.021	0.155

^A In samples designated A one side was extracted, while in samples designated B the other side was extracted.

^B N.A. not analyzed.