

**Analysis of Polychlorinated Biphenyls (PCBs) in Water
Samples Collected February 28 – March 1, 2000
from the Bayou Creek System**

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INTRODUCTION

Samples were taken for PCB assays from the water column of Big and Little Bayou Creeks on February 28 and March 1, 2000. Sampling stations BB1 through BB9 on Big Bayou Creek and LB1 through LB4 on Little Bayou Creek were included in this survey. An additional reference station, upstream of BB1 and designated BB1A, also was collected. In addition, Massac Creek (MC) was sampled and served as a reference station independent of the Bayou Creek system. Two samples were collected for PCB assays for each of the sampling stations. Three Aroclors (*i.e.* 1248, 1254, and 1260) were determined for the 32 samples.

METHODS

Water Collection

Water samples for PCB analyses were collected in I-Chem chemically cleaned, 1-L amber glass jars with teflon-lined caps. Samples for PCB determinations were then sealed and placed on ice until delivery to the laboratory and maintained under refrigeration (4°C) until extraction.

Water Extraction

Liquid-liquid extractions were performed in separatory funnels following SW-846 Method 3510C, Separatory funnel liquid-liquid extraction (U.S. EPA, 1997). Water samples were extracted within 7 days of collection. One-liter aqueous samples were extracted three times with 60-mL methylene chloride and concentrated to near dryness in a Roto-evaporator (Buchi Model RE121). Reconstituted samples (2.0 mL in iso-octane) were cleaned of interferences as described below and then analyzed by gas chromatography.

PCB Sample Cleanup

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 hexane and evaporated to 2.0 mL (Erickson, 1997; U.S. EPA, 1997, 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, 1997, SW-846 Method 3660B, Sulfur cleanup). A 4 µL sub-sample was then analyzed by gas chromatography.

PCB Determinations

Samples were analyzed for Aroclors 1248, 1254, and 1260 according to SW-846 Method 8082 (Polychlorinated biphenyls by gas chromatography; U.S. EPA, 1997). Analyses were 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 (0.5µm 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. Aroclor concentrations 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. Statistical quantitation of peak heights was determined by multiple-peak linear regression analysis, which was performed with Lotus-123® software. 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). Chromatographs and bench records for all PCB assays will be maintained as given below under quality assurance.

Quality Assurance

Permanent bench records were kept of all assays and annotated as required under Good Laboratory Practices (Federal Register, 40 CFR, Part 160, August 17, 1989). All printouts and graphic recordings were filed and are open for inspection. These bench records will be archived within two years after the close of the project but retrievable upon request. Chain of Custody were maintained for all samples collected.

RESULTS

As noted above, assays were conducted for Aroclors 1248, 1254, and 1260 for 32 water samples from the Bayou Creek system (Tables 1, 2). There was no detection of PCBs at a detection limit of 0.08 µg/L (*i.e.* 80 ppt). It should be noted, however, that the residence time (half-life) of most PCB congeners is quite short, due to rather rapid partitioning into the sediments and biomass, as well as some loss to the atmosphere. While these results presented in Tables 1 and 2 are encouraging, it is recommended that routine monitoring be continued, due to the persistence of PCB residues in fish and wildlife in and around the Bayou Creek system (Birge and Price, 2001; Birge *et al.* 1998; Price and Birge, 1998a, 1998b). Assays of controls and recoveries are given in Tables 3 and 4. No procedural contamination was noted and PCB recoveries ranged from 90% to 114%.

Table 1. PCB results for surface water samples collected from Big Bayou Creek, February 29 and March 1, 2000.

Sampling Station	Aroclor Concentration ($\mu\text{g/L}$)		
	1248	1254	1260
MC-30100-PWS1	<0.082	<0.082	<0.082
MC-30100-PWS2	<0.081	<0.081	<0.081
BB1A-22900-PWS1	<0.081	<0.081	<0.081
BB1A-22900-PWS2	<0.081	<0.081	<0.081
BB1-22900-PWS1	<0.083	<0.083	<0.083
BB1-22900-PWS2	<0.082	<0.082	<0.082
BB2-30100-PWS1	<0.080	<0.080	<0.080
BB2-30100-PWS2	<0.081	<0.081	<0.081
BB3-30100-PWS1	<0.081	<0.081	<0.081
BB3-30100-PWS2	<0.086	<0.086	<0.086
BB4-30100-PWS1	<0.083	<0.083	<0.083
BB4-30100-PWS2	<0.081	<0.081	<0.081
BB5-30100-PWS1	<0.082	<0.082	<0.082
BB5-30100-PWS2	<0.082	<0.082	<0.082
BB6-30100-PWS1	<0.082	<0.082	<0.082
BB6-30100-PWS2	<0.082	<0.082	<0.082
BB7-22900-PWS1	<0.082	<0.082	<0.082
BB7-22900-PWS2	<0.082	<0.082	<0.082
BB8-22900-PWS1	<0.080	<0.080	<0.080
BB8-22900-PWS2	<0.081	<0.081	<0.081
BB9-30100-PWS1	<0.082	<0.082	<0.082
BB9-30100-PWS2	<0.089	<0.089	<0.089

Table 2. PCB results for surface water samples collected from Little Bayou Creek, February 29 and March 1, 2000.

Sampling Station	Aroclor Concentration ($\mu\text{g/L}$)		
	1248	1254	1260
LB1-30100-PWS1	<0.083	<0.083	<0.083
LB1-30100-PWS2	<0.082	<0.082	<0.082
LB2-30100-PWS1	<0.091	<0.091	<0.091
LB2-30100-PWS2	<0.089	<0.089	<0.089
LB2A-30100-PWS1	<0.082	<0.082	<0.082
LB2A-30100-PWS2	<0.082	<0.082	<0.082
LB3-30100-PWS1	<0.087	<0.087	<0.087
LB3-30100-PWS2	<0.084	<0.084	<0.084
LB4-30100-PWS1	<0.081	<0.081	<0.081
LB4-30100-PWS2	<0.082	<0.082	<0.082

Table 3. PCB Concentrations in Controls^a used in analysis of Water Samples from Bayou Creek System, Collected February 29 and March 1, 2000.

Sample Name	Aroclor Conc. (µg/L)		
	1248	1254	1260
CON-030800-PCON1W	<0.080	<0.080	<0.080
CON-030800-PCON2W	<0.080	<0.080	<0.080

^a 1.0 L deionized water extracted as a regular sample.

Table 4. PCB Concentrations in Procedure Recoveries^a for Water Samples from Bayou Creek System Collected February 29 and March 1, 2000.

Sample Name	Aroclor	µg Spike	Aroclor Conc. (µg)		Percent Recovery
			Expected Conc.	Measured Conc.	
RECO-030900-PPRR1	1248	1.0	1.000	0.896	90
RECO-030900-PPRR2	1254	1.0	1.000	1.14	114
RECO-030900-PPRR3	1260	1.0	1.000	0.935	94

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

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