Lake Champlain Research Institute State University of New York, Plattsburgh

Cumberland Bay PCB Study

Appendix B:
Water Column PCB Analysis
Quality Assurance and Data Report

Submitted to
U.S. Envrionmental Protection Agency
And
Lake Champlain Basin Program

by

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Cumberland Bay PCB Project

Quality Assurance Document And Data Report

SUNY Plattsburgh Subcontract

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Project Overview

The objective of this phase of the Cumberland Bay PCB Project was to determine Polychlorinated Biphenyl (PCB) congener concentrations in water samples from Cumberland Bay and the Main Lake portion of Lake Champlain, New York. Large volume water samples were collected from nine different sites from 2-4 times through the summer of 1996, representing 25 samples (50 analyses) in the particulate and dissolved phases. Samples were filtered and extracted onto XAD – 2 resin, soxhlet extracted, concentrated and analyzed by gas chromatography with electron capture detection following protocols established through the Great Lakes Research Programs (Swackhamer, 1988). This QA summary includes data on concentrations of 90 congeners measured in those analyses as well as information summarizing the precision and accuracy of these procedures.

Methods

Sample detection limits on the order of 0.1 ng/L (0.1 pptr) total PCB and 5 pg/L for individual congeners are necessary for most water analyses. This requires ultra clean sampling and analytical methods and substantial sample pre-concentration. Procedures to do low-level PCB analysis have been developed and documented in U.S.E.P.A. sponsored projects in the Great Lakes and have recently been used in other areas (Swackhamer and Armstrong, 1987; Swackhamer, 1988; Jeremiason et al., 1994; Verbrugge et al., 1995).

Sampling

A submersible stainless steel pump was used to collect 160 liters of water in rigorously cleaned 20L glass carboys aboard the SUNY Plattsburgh Research Vessel Monitor. Samples were immediately returned to the lab, filtered and extracted onto XAD-2 resin within 24 hours of sample collection.

Sample Extraction and Processing

Water samples were filtered with an all Teflon pump through a stainless steel monoplate with a 30cm diameter ashed glass fiber filter to remove suspended sediments for PCB extraction. Filters were ashed at 450°C for 16 hours prior to use. The aqueous phase was pumped through a hydrophobic XAD-2 resin (90-110 ml) which quantitatively retains PCBs (Swackhamer and Armstrong, 1987). Flow rates were limited to 5 bed volumes per minute. XAD-2 Resin (Sigma Chemical 9060-05-3; 20-60 mesh) was cleaned in a large volume (3,000ml solvent capacity/1 kg XAD) soxhlet extractor using the procedure of Jeremiason et al. (1994). The resin was first rinsed with 2-3 liters of Type I Milli-Q water to remove salts, followed by soxhlet extraction for 24 hrs. each with the following solvents: methanol, acetone, hexane, methylene chloride, hexane, acetone and methanol. Methylene chloride extraction ensures maximum swelling of the resin to extract entrained monomers.

The XAD resin containing aqueous phase PCBs and the filter containing the particulate phase were soxhlet extracted separately into approximately 300 ml of 50:50 acetone/hexane. All pesticide grade solvents used after this point were concentrated and analyzed for ECD detectable impurities before use. Extracts were transferred to separatory funnels where the water phase was separated and back-extracted with hexane. The hexane fractions were combined and back-extracted with purified H₂O to remove the bulk of acetone and polar compounds from the sample. Extracts were concentrated using a Rotary Evaporator to approximately 5 mls and transferred to a liquid/solid chromatography column containing Na₂SO₄, deactivated alumina and deactivated silica for cleanup. Primarily nonpolar sample components were eluted with approximately 100 ml hexane which was further concentrated down to 3-5 ml by rotary evaporation. Dry N₂ blowdown was used to give a final sample volume of 0.5 ml. Aliquots of the final sample were transferred into 200 µl glass autosampler vials with Teflon crimp-top septa. Samples were stored in a freezer at -20°C and run within one day.

Analytical Procedure

Samples were analyzed on a HP 5890 Gas Chromatograph with electron capture detectors, electronic pressure control, and an HP7673 autosampler. Congeners were separated on a 50m HP5 capillary column (Table 1). This column has been used for congener analysis for many years and congener elution patterns are well characterized (Mullin, 1985; Swackhamer, 1988). Chromatographic conditions are given in Table 1.

Table 1. Chromatographic Conditions for PCB Congener Separation.

<u>Temper</u>	ratu	re Program	Chromatographic (Cond	tions
Initial Temp) -	80° for 2 min.	Carrier Gas	-	Hydrogen
Ramp 1	-	5°/min to 160	Carrier Linear Velocity	-	45cm/sec.
-	_	1°/min to 260	Makeup Gas	-	Ar/Methane
Ramp3	_	10°/min to 290	Injection mode	-	Splitless
Final Time	_	5° min @ 290	Splitless purge flow	-	60 ml/min.
Total time	_	126 minutes.	Splitless purge delay	-	1.7 min.
		•			

Congener Identification. Two approaches were used to identify individual congeners. In the first, approximately 100 individual congeners (supplied by Ultra Scientific or Accustandards) were purchased individually and mixed in stocks of 20 congeners per solution so that each congener was fully resolved (New York State DOH, 1991). Five stock solutions were prepared and diluted into standards. In the second approach, a mixture of three Aroclors (1232:1248:1262 at a 25:18:18 ratio), previously characterized to the congener level (Mullin, 1985; Swackhamer, 1988) was produced at a total PCB concentration of 610 ng/L and will be referred to as the 610 ng/L performance standard. The latter procedure has been used in several recent studies (Hornbuckle et al., 1993; Verbrugge et al., 1995) for congener quantitation, so the first procedure was used as a check for congener identification. All standards, samples, blanks, duplicates and matrix spike solutions were spiked with one ml of a surrogate spike solution consisting of congeners #14, #65 and #166 at concentrations of 20, 8 and 6 ng/ml, respectively, immediately

following filtration. Immediately prior to final N_2 blowdown, samples were spiked using congeners #30 and #204 to serve as internal standards and to determine relative retention times for congener identification.

Congener Quantification

Relative retention times were calculated by comparison to the internal standard #204. Congeners were identified by comparison of relative retention time in the sample to relative retention time in the 610 ng/ml performance standard. For correct identification, retention time of a congener must be within a window of ±3 sec. of the predicted retention time based on correction using relative retention. Sample concentrations were not corrected for analytical losses, but losses were calculated for each sample using surrogate spike recovery and were found to be minimal. Relative Response Factors (RRF) were calculated in the performance standard for each congener using the following formula:

$$RRF_{std} = \left(\frac{mass\ congener}{area\ congener}\right)_{std} \div \left(\frac{mass\ istd}{area\ istd}\right)_{std}$$

RRFs for congeners eluting prior to #110 were computed using internal standard #30 response. RRFs for congeners eluting after #82 were quantified using #204. Average RRFs were determined using standards in the range of the sample concentrations, since retention time can vary with concentration (Swackhamer, 1988). Congener concentrations in the sample were calculated using the following equation:

$$(\text{conc. congener})_{\text{sample}} = \frac{(\text{area congener})_{\text{sample}} \times \text{RRF}_{\text{std}} \times \left(\frac{\text{mass istd}}{\text{area istd}}\right)}{(\text{volume of sample collected})}$$

Study Sites

Water samples were collected from shore or from the SUNY Plattsburgh research vessel

Monitor throughout the summer and fall of 1996 from the Saranac River, across a transect of sites

in Cumberland Bay designed to distinguish inputs from Wilcox Dock, and at four Main Lake sites from Four Brothers Islands in the south to a site west of the Gut in the North. Samples were collected from approximately two thirds of maximum depth in shallow bay sites. In deeper Main Lake sites a composite sample of equal volumes from 5 and 22 m deep was taken. Since only two samples could be processed per week, samples were taken in small batches to meet necessary holding times. Exact locations and depths of each sample site are given in Table 2.

Table 2. Latitudes and longitudes of sample sites used in this study.

Site	Latitude	Longitude	Site Depth (m)	Sample depths
Saranac River	44°41.88'	73°27.08'	1	0.5m
Cumberland Ba	y Sites			
B5	44°42.86'	73°26.15'	3	2.0 m
B12	44°42.43'	73°25.01'	11	6.7 m
B15	44°42.05'	73°26.14'	4	2.4 m
B30	44°41.20'	73°25.04'	15	9.0 m
Main Lake Site	S			
B45	44°40.35'	73°23.32'	36	5m/22 m composite
Stave Island	44°31.67'	73°18.97'	42	5m/22 m composite
Four Brothers	44°24.84'	73°19.72'	43	5m/22 m composite
The Gut	44°45.37	73°21.30	50	5m/22 m composite

Quality Assurance Results

Procedural Blanks and Blank Correction

Four procedural blank samples, consisting of 160L of XAD-cleaned, Type I deionized water were processed with every sample set. Each procedural blank was processed in a manner identical to that for samples. Figure 1 shows an average of these four blanks. Most of the PCB contamination of the blanks was limited to mid-range congeners which are not characteristic of most Lake Champlain water samples collected in this study.

The congener pattern evident in the blank was also found in all other samples, indicating that blank contamination did not arise from the blank water itself, but rather from contamination during portions of the procedure common to both blanks and samples. A thorough investigation

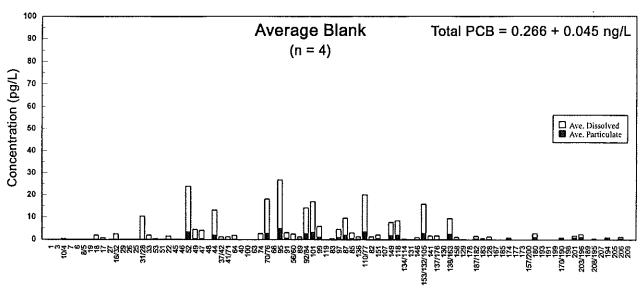


Figure 1. PCB congener distribution in an average of four procedural blanks consisting of 160 L Of XAD cleaned milli-q water.

of the analytical procedure indicated that the laboratory/building atmosphere was the source of contamination which occurred largely during soxhlet extraction.

To avoid bias arising from sample contamination, and since sample contamination was evident in the congener pattern in each sample, the step was taken to subtract the average blank concentration (Table 3) from each sample for each congener. Under optimum conditions, such blank correction would be done using a unique blank processed with each sample. Cost considerations made this impossible. However, since all of the blanks were fairly consistent (Table 3 and Figure 2), using the average blank would only introduce minor errors. Most of the congeners in the blanks were in the middle portion of the chromatogram. Consequently, early eluting congeners characteristic of Aroclor 1242 and late eluting congeners characteristic of Aroclor 1260 were not affected. To account for the small variability encountered in the blanks, detected congeners were flagged if their concentrations were less than the standard deviation of the average blank. Although these flagged congeners are included in the sample to avoid introducing bias, eliminating them would produce only small changes in sample concentrations (<10% in most cases).

Total PCB concentrations in the four procedural blanks were very consistent, both for total PCBs and individual congeners, averaging 0.266 ± 0.045 ng/L of total PCB. Of the 88 congeners quantified, 74 had average blank concentrations less than 5 pg/L. Seventy eight of the 88 congeners had a blank variability less than 2 pg/L. The congener pattern for the the individual blanks (Figure 2) was also consistent.

Table 3. Congener concentrations and variability for the four procedural blanks collected during Lake Champlain sampling in 1996. All concentrations are in pg/L except total PCB (last row) which is in ng/L.

Congener	June	17	Jul	y 8	July 2	2	Augu	st 15	Average	Blank	Blank Va	riation
BZ#	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.
1	0	0	0	0	0	0	0	0	0	0	0.00	0.00
3	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
10/4	0	0	0.74	0	0.73	0	0	0	0.37	0.00	0.42	0.00
7	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
6	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
19	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
18	3.47	0	0.73	0	2.05	0	1.61	0	1.97	0.00	1.14	0.00
17	0	0	0.91	0	2.22	0	0	0	0.78	0.00	1.05	0.00
27	0	0	0	0	. 0	0	0	0	0.00	0.00	0.00	0.00
16/32	1.81	0	1.72	0	3.44	0	2.98	0	2.49	0.00	0.86	0.00
29	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
26	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
25	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
31/28	6.21	0	6.89	0	12.85	0	15.43	0	10.35	0.00	4.52	0.00
33	0	0	1.32	0	3.01	0	3.38	0	1.93	0.00	1.57	0.00
53	0	0	0	0	0.79	0	1	0	0.45	0.00	0.52	0.00
51	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
22	0	0	1.6	0	1.98	0	2.14	0	1.43	0.00	0.98	0.00
45	0	0	0	0	0.41	0	0	0	0.10	0.00	0.21	0.00
46	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
52	18.07	2.81	18.2	2.42	20,99	4.54	24.95	3.75	20.55	3,38	3.23	0.95
49	3.59	0	3.56	0	5.06	0.74	4.95	0	4.29	0.19	0.83	0.37
47	2.71	0	2.81	0	6.04	1.42	3.3	0	3.72	0.36	1.57	0.71
48	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
44	9.6	1.71	9.99	1.14	10.79	2.82	14.25	2.24	11.16	1.98	2.12	0.72
37/42	0.99	0	0.85	0	1.17	0	1.88	0	1.22	0.00	0.46	0.00
41/71	0	0	1.19	0	1.44	0	2.09	0	1.18	0.00	0.87	0.00
64	1.46	0	1.62	0	1.72	0.38	2.47	0	1.82	0.10	0.45	0.19
40	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
100	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
63	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
74	2.66	0	2.45	0	2.34	0	3.48	0	2.73	0.00	0.52	0.00
70/76	14.52	2.47	14.58	1.94	13.35	3.25	19.12	3.56	15.39	2.81	2.55	0.74
66	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
95	19.4	3.76	21.93	3.27	19.14	5.5	27.56	6.73	22.01	4.82	3.91	1.60
91	2.26	0.43	2.78	0	2.5	0.71	3.2	0.78	2.69	0.48	0.40	0.35
56/60	2.6	0	2.19	0	2.04	0	3.23	0	2.52	0.00	0.53	0.00
89	0.94	0.16	1.03	0.15	0.86	0.25	1.24	0.3	1.02	0.22	0.16	0.07
92/84	9.66	1.89	12.44	1.85	9.87	3.08	13.91	3.87	11.47	2.67	2.06	0.98
101	13.38	2.88	14.25	2.3	11.37	3.55	16.21	4.16	13.80	3.22	2.01	0.81
99	4.51	0.9	5.1	0.77	4.05	1.2	5.81	1.38	4.87	1.06	0.76	0.28
119	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
83	0.5	0	0.72	0	0.35	0	0.53	0	0.53	0.00	0.15	0.00
97	3.26	0.69	4.28	0.64	2.88	1.02	4.17	1.19	3.65	0.89	0.69	0.26
87	6.8	1.69	8.46	1.62	6	2.23	8.83	2.6	7.52	2.04	1.35	0.46
85	2.41	0.46	2.67	0	2.11	0.66	2.98	0.75	2.54	0.47	0.37	0.33
136	1.2	0	1.21	0	0.9	0.31	1.31	0.4	1.16	0.18	0.18	0.21
110/77	15.25	1.26	20.09	3	12.4	4.08	19.22	5.24	16.74	3.40	3.58	1.69
82	1.15	0	1.62	0	1.09	0	1.04	0.37	1.23	0.09	0.27	0.19

Table 3. (continued) Congener concentrations and variability for the four procedural blanks collected during Lake Champlain sampling in 1996. All concentrations are in pg/L except total PCB (last row) which is in ng/L.

Congener	June		July	8	July	22	August	15	Average	Blank	Blank Va	riation
BZ#	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.
151	2.03	0.44	1.94	0	1.56	0.51	1.4	0.58	1.73	0.38	0.30	0.26
107	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
149	6.5	1.93	6.49	1.25	4.51	1.87	5.81	2.22	5.83	1.82	0.94	0.41
118	6.67	2.25	7.31	1.45	5.16	1.78	7.04	1.96	6.55	1.86	0.96	0.33
134/114	0	0	0	0	0	0	0.93	0	0.23	0.00	0.47	0.00
131	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
146	1.03	0	1.3	0	0.6	0	0.8	0	0.93	0.00	0.30	0.00
153/132/105	10.78	5.04	16.83	1.59	11.13	1.85	14.08	2.35	13.21	2.71	2.83	1.59
141	1.61	0	2.04	0	1.3	0	1.55	0	1.63	0.00	0.31	0.00
137/176	0	0	7	0	0	0	0	0	1.75	0.00	3.50	0.00
130	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
138/163	8.19	2.72	8.13	1.85	4.88	2.24	6.65	2.93	6.96	2.44	1.56	0.49
158	1.24	0	1.29	0	0.76	0	1.04	0	1.08	0.00	0.24	0.00
129	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
178	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
187/182	1.05	0	1.1	0	1.42	0.66	1.24	0.84	1.20	0.38	0.17	0.44
183	0	0	0.69	0	0.66	0	0.61	0	0.49	0.00	0.33	0.00
128	0.92	0	2.76	0	0.66	0	0.94	0	1.32	0.00	0.97	0.00
167	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
185	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
174	0.74	0	0.49	0	0.68	0.34	0.52	0.53	0.61	0.22	0.12	0.26
177	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
173	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
157/200	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
180	1.67	0	0	0.57	2.91	1.11	2.41	2.35	1.75	1.01	1.27	1.00
193	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
191	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
199	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
170/190	0	0	0.9	0	0.87	0	0.8	0.88	0.64	0.22	0.43	0.44
198	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
201	0	0	0	0	2.26	1.02	1.45	1.95	0.93	0.74	1.12	0.94
203/196	0	0	0	0	3.34	1.53	2	2.41	1.34	0.99	1.64	1.19
189	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
208/195	0	0	0	0	1.37	0	0	0	0.34	0.00	0.69	0.00
207	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
194	0	0	0.33	0	1.27	0.52	0.82	1.06	0.61	0.40	0.56	0.51
205	0	0	0	0	0	0	0	0	0.00	0.00	0.00	0.00
206	0	0	0.39	0	1.63	0.57	1.06	1.24	0.77	0.45	0.72	0.59
209	0	0	0.16	0.14	0.16	0	0.15	0	0.12	0.04	0.08	0.07
Total PCB	June		July	8	July	22	August		Average		Blank Va	riation
	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.
pg/L	190.8	33,49	227.1	26.00	213.1	49.7	263.6	58.62	223.6	41.95	30.52	14.90
ng/L		0.224		0.253		0.263		0.322		0.266		0.045

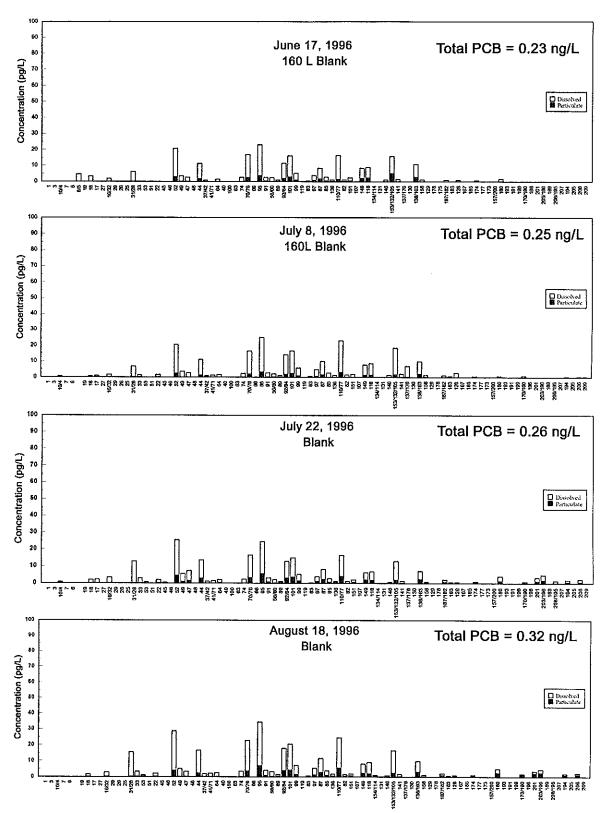


Figure 2. Congener pattern for the four 160L procedural blanks collected during lake sampling.

Precision (duplicate analysis)

Precision was assessed by measurement of duplicate 160L water samples collected simultaneously from the same site. Figures 2 and 3 and Tables 4 and 5 document the results for two duplicate samples taken from site B12. The overall congener pattern (Figs. 2 and 3) for the duplicates was very similar. Precision, expressed as relative standard deviation (100 x S.D./Mean), for total PCBs was 18.3% and 5.1% for the particulate fraction and 9.8% and 10.3% for the dissolved fraction in the two samples. The combined fractions for total PCBs had 12.3% and 7.6% RSD. These values are typical for environmental analyses with large amounts of sample manipulation including extraction and concentration (e.g. Swackhamer, 1988)

Precision for individual congeners was only determined when both congeners were detected, since at these low levels a congener may only be detected in one of the duplicates if it is near the detection limit. For the particulate phase precision averaged 18.9±11.1% and 10.3±11.0% RSD for the two duplicate samples. For the dissolved phase precision averaged 26.5±26.9% and 14.5±14.7% RSD for the two samples.

Control Charts. Instrument precision and reproducibility can also be assured by repeated measurements of QA samples during each run. Unfortunately, low level aqueous QA samples for PCBs are not available. Each sample, however, is spiked with a surrogate mix of three congeners (14, 65 and 166), and the original spiking solution is run with each sample to measure surrogate spike recovery. Fig. 4 shows response factors for these three congeners over the time of the investigation and documents the reproducibility of instrument response. Every sample is also run with the 610 standard, which must have congeners identified and response factors quantified. We routinely use the old standard to identify congeners in the new standard, and at the same time compare both retention times and response factors between the old and new standards. In all cases, new response factors were within 20% of the old response factors for all 88 peaks identified.

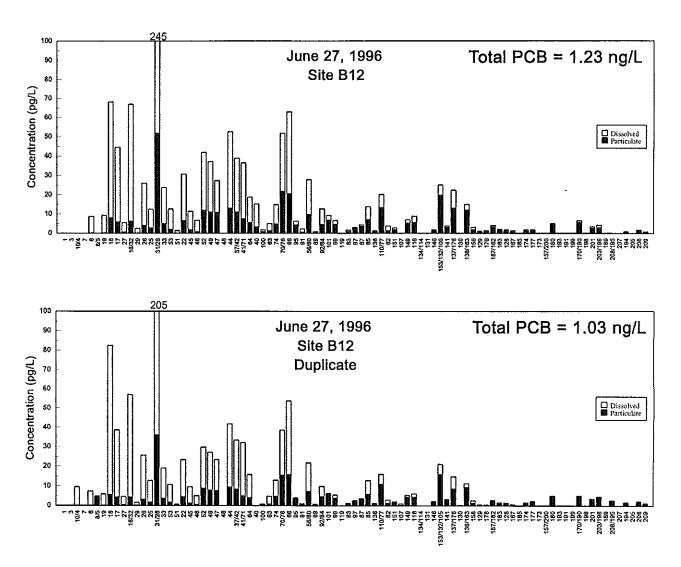
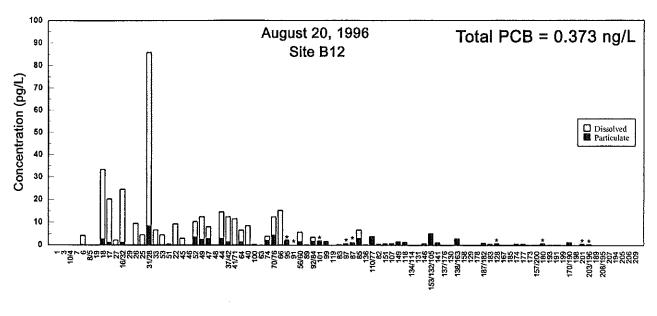


Figure 3. PCB congener concentrations in the particulate and dissolved phases of duplicate samples collected simultaneously from site B12 on June 26, 1996.



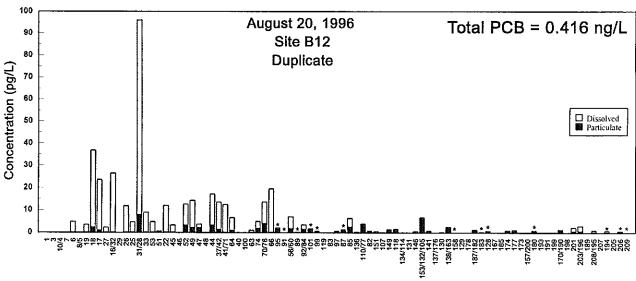


Figure 4. PCB congener concentrations in the particulate and dissolved phases of duplicate samples collected simultaneously from site B12 on August 20, 1996.

Table 4. Precision expressed as RSD for congeners and total PCB based on measurement of duplicate samples from site B12 on June 27, 1996. Units are in pg/L.

BZ#							···	its are in pa	· · · · · · · · · · · · · · · · · · ·		
1	Congener									%RSD	%RSD
3										Part.	Diss.
10/4			• • • • • • • • • • • • • • • • • • • 								<u> </u>
7 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 6 0.00 8.84 0.00 7.31 0.00 8.08 0.00 1.08 13 19 0.00 9.24 0.00 5.92 0.00 7.58 0.00 2.35 30 18 8.08 60.15 5.45 77.10 6.77 68.62 1.86 11.99 22.74 9.77 17 5.82 38.80 4.21 34.41 5.02 36.60 1.14 3.10 22.70 8.7 16/32 6.19 60.78 4.20 52.95 5.20 56.87 1.41 5.54 227.99 9.9 29 0.00 2.63 0.00 1.58 0.00 2.11 0.00 0.74 7.35 2.79.99 9.9 26 4.09 21.91 3.03 22.53 3.56 22.22 0.75 0.44 21.05 11 <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td>1</td></t<>											1
6											<u>i</u>
19											-
18											13.40
17 5.82 38.80 4.21 34.41 5.02 36.60 1.14 3.10 22.70 8.											30.97
27										27.49	17.47
16/32										22.70	8.48
29 0.00 2.63 0.00 1.58 0.00 2.11 0.00 0.74 35 26 4.09 21.91 3.03 22.53 3.56 22.22 0.75 0.44 21.05 1.1 25 2.75 9.89 1.58 11.22 2.17 10.56 0.83 0.94 38.21 8. 31/28 51.70 193.27 35.95 169.26 43.83 181.26 11.14 16.98 25.41 9. 33 4.90 18.90 3.59 15.44 4.25 17.17 0.93 2.45 21.82 14 53 2.02 10.67 1.63 9.18 1.83 9.93 0.28 1.05 15.11 10.00 51 0.00 1.46 0.00 0.66 0.00 1.06 0.00 0.57 53 45 1.80 9.60 1.23 8.32 1.52 8.96 0.40 0.91 26.60 10.0 <td></td> <td>10.13</td>											10.13
26 4.09 21.91 3.03 22.53 3.56 22.22 0.75 0.44 21.05 1. 25 2.75 9.89 1.58 11.22 2.17 10.56 0.83 0.94 38.21 8.3 31/28 51.70 193.27 35.95 169.26 43.83 181.26 11.14 16.98 25.41 9.2 33 4.90 18.90 3.59 15.44 4.25 17.17 0.93 2.45 21.82 14. 53 2.02 10.67 1.63 9.18 1.83 9.93 0.28 1.05 15.11 10. 51 0.00 1.46 0.00 0.66 0.00 1.06 0.00 0.57 53. 42 6.39 24.39 4.47 18.91 5.43 21.65 1.36 3.87 25.00 15.11 10. 45 1.80 9.60 1.23 8.32 1.52 8.96 0.40 0.91										27.09	9.74
25 2.75 9.89 1.58 11.22 2.117 10.56 0.83 0.94 38.21 8.3 31/28 51.70 193.27 35.95 169.26 43.83 181.26 11.14 16.98 25.41 9.2 33 4.90 18.90 3.59 15.44 4.25 17.17 0.93 2.45 21.82 14 53 2.02 10.67 1.63 9.18 1.83 9.93 0.28 1.05 15.11 10. 51 0.00 1.46 0.00 0.66 0.00 1.06 0.00 0.57 53 22 6.39 24.39 4.47 18.91 5.43 21.65 1.36 3.87 25.00 17 45 1.80 9.60 1.23 8.32 1.52 8.96 0.40 0.91 26.60 10. 46 0.69 6.09 0.51 4.57 0.60 5.33 0.13 1.07 21.21									0.74		35.27
S1/28									0.44	21.05	1.97
33 4.90 18.90 3.59 15.44 4.25 17.17 0.93 2.45 21.82 14. 53 2.02 10.67 1.63 9.18 1.83 9.93 0.28 1.05 15.11 10 51 0.00 1.46 0.00 0.66 0.00 1.06 0.00 0.57 53 22 6.39 24.39 4.47 18.91 5.43 21.65 1.36 3.87 25.00 17 45 1.80 9.60 1.23 8.32 1.52 8.96 0.40 0.91 26.60 10 46 0.69 6.09 0.51 4.57 0.60 5.33 0.13 1.07 21.21 20 52 11.85 30.09 8.69 21.10 10.27 25.59 2.23 6.36 21.76 24. 49 11.05 26.20 7.86 19.35 9.45 22.78 2.26 4.84 23.87 21.								0.83	0.94	38.21	8.91
53 2.02 10.67 1.63 9.18 1.83 9.93 0.28 1.05 15.11 10 51 0.00 1.46 0.00 0.66 0.00 1.06 0.00 0.57 53 22 6.39 24.39 4.47 18.91 5.43 21.65 1.36 3.87 25.00 17 45 1.80 9.60 1.23 8.32 1.52 8.96 0.40 0.91 26.60 10 46 0.69 6.09 0.51 4.57 0.60 5.33 0.13 1.07 21.21 20 52 11.85 30.09 8.69 21.10 10.27 25.59 2.23 6.36 21.76 24 49 11.05 26.20 7.86 19.35 9.45 22.78 2.26 4.84 23.87 21. 47 10.70 16.71 7.48 15.99 9.09 16.35 2.28 0.51 25.06 3.<								11.14	16.98	25.41	9.37
51 0.00 1.46 0.00 0.66 0.00 1.06 0.00 0.57 53 22 6.39 24.39 4.47 18.91 5.43 21.65 1.36 3.87 25.00 17 45 1.80 9.60 1.23 8.32 1.52 8.96 0.40 0.91 26.60 10 46 0.69 6.09 0.51 4.57 0.60 5.33 0.13 1.07 21.21 20 52 11.85 30.09 8.69 21.10 10.27 25.59 2.23 6.36 21.76 24 49 11.05 26.20 7.86 19.35 9.45 22.78 2.26 4.84 23.87 21. 47 10.70 16.71 7.48 15.99 9.09 16.35 2.28 0.51 25.06 3. 48 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00<								0.93	2.45	21.82	14.25
22 6.39 24.39 4.47 18.91 5.43 21.65 1.36 3.87 25.00 17 45 1.80 9.60 1.23 8.32 1.52 8.96 0.40 0.91 26.60 10 46 0.69 6.09 0.51 4.57 0.60 5.33 0.13 1.07 21.21 20 52 11.85 30.09 8.69 21.10 10.27 25.59 2.23 6.36 21.76 24 49 11.05 26.20 7.86 19.35 9.45 22.78 2.26 4.84 23.87 2.1 47 10.70 16.71 7.48 15.99 9.09 16.35 2.28 0.51 25.06 3 48 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 44 12.95 39.57 9.40 32.50 11.18 36.04 2.51 5.00 22.46 13								0.28	1.05	15.11	10.61
45 1.80 9.60 1.23 8.32 1.52 8.96 0.40 0.91 26.60 10 46 0.69 6.09 0.51 4.57 0.60 5.33 0.13 1.07 21.21 20 52 11.85 30.09 8.69 21.10 10.27 25.59 2.23 6.36 21.76 24 49 11.05 26.20 7.86 19.35 9.45 22.78 2.26 4.84 23.87 21 47 10.70 16.71 7.48 15.99 9.09 16.35 2.28 0.51 25.06 3. 48 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 44 12.95 39.57 9.40 32.50 11.18 36.04 2.51 5.00 22.46 13. 37/42 10.92 28.00 8.39 25.13 9.66 26.56 1.79							1.06	0.00	0.57		53.37
46 0.69 6.09 0.51 4.57 0.60 5.33 0.13 1.07 21.21 20 52 11.85 30.09 8.69 21.10 10.27 25.59 2.23 6.36 21.76 24 49 11.05 26.20 7.86 19.35 9.45 22.78 2.26 4.84 23.87 21. 47 10.70 16.71 7.48 15.99 9.09 16.35 2.28 0.51 25.06 3. 48 0.00 1.18 36.04 2.51 5.00 22.46 13. 37.42 1.01 3.33								1.36	3.87	25.00	17.90
52 11.85 30.09 8.69 21.10 10.27 25.59 2.23 6.36 21.76 24. 49 11.05 26.20 7.86 19.35 9.45 22.78 2.26 4.84 23.87 21. 47 10.70 16.71 7.48 15.99 9.09 16.35 2.28 0.51 25.06 3. 48 0.00 1.61 6.05 2.28 8.55 1 100 0.72 1.17 0.00 0.72 1.161 6.05 2.28 8.55 1 100 0.72 1.17 0.00								0.40	0.91	26.60	10.10
49 11.05 26.20 7.86 19.35 9.45 22.78 2.26 4.84 23.87 21. 47 10.70 16.71 7.48 15.99 9.09 16.35 2.28 0.51 25.06 3. 48 0.00 1.03 1.08 21.77 1.38 28.79 4.8 4.4 4.71 12.63 1.03 1.08 21.77 8.5 4.64 5.44 13.39 3.99 11.86 4.71 12.63 1.03 1.08 21.77 8.5 4.0 3.22 12.09 0.00 0.00 1.61 6.05 2.28 8.55 8.5 1						0.60		0.13	1.07	21,21	20.17
47 10.70 16.71 7.48 15.99 9.09 16.35 2.28 0.51 25.06 3. 48 0.00 0.0						10.27	25.59	2.23	6.36	21.76	24.84
48 0.00 11.18 36.04 2.51 5.00 22.46 13.3 37/42 10.02 28.00 8.39 25.13 9.66 26.56 1.79 2.03 18.53 7.0 41/71 7.39 29.19 4.89 27.24 6.14 28.22 1.77 1.38 28.79 4.8 64 5.44 13.39 3.99 11.86 4.71 12.63 1.03 1.08 21.77 8.3 40 3.22 12.09 0.00 0.00 1.61 6.05 2.28 8.55 10.00 10.00 10.00 10.00						9.45	22.78	2.26	4.84	23.87	21.27
44 12.95 39.57 9.40 32.50 11.18 36.04 2.51 5.00 22.46 13. 37/42 10.92 28.00 8.39 25.13 9.66 26.56 1.79 2.03 18.53 7.0 41/71 7.39 29.19 4.89 27.24 6.14 28.22 1.77 1.38 28.79 4.8 64 5.44 13.39 3.99 11.86 4.71 12.63 1.03 1.08 21.77 8.3 40 3.22 12.09 0.00 0.00 1.61 6.05 2.28 8.55 100 0.72 1.17 0.00 0.72 0.36 0.95 0.51 0.32 33. 63 1.24 3.89 0.79 4.05 1.02 3.97 0.32 0.11 31.35 2.8 70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18. <tr< td=""><td></td><td></td><td>-</td><td></td><td></td><td>9.09</td><td>16.35</td><td>2.28</td><td>0.51</td><td>25.06</td><td>3.11</td></tr<>			-			9.09	16.35	2.28	0.51	25.06	3.11
37/42 10.92 28.00 8.39 25.13 9.66 26.56 1.79 2.03 18.53 7.6 41/71 7.39 29.19 4.89 27.24 6.14 28.22 1.77 1.38 28.79 4.8 64 5.44 13.39 3.99 11.86 4.71 12.63 1.03 1.08 21.77 8.5 40 3.22 12.09 0.00 0.00 1.61 6.05 2.28 8.55 100 0.72 1.17 0.00 0.72 0.36 0.95 0.51 0.32 33. 63 1.24 3.89 0.79 4.05 1.02 3.97 0.32 0.11 31.35 2.8 74 4.76 10.20 4.61 8.29 4.69 9.24 0.11 1.35 2.26 14. 70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18.					0.00	0.00	0.00	0.00	0.00		l l
37/42 10.92 28.00 8.39 25.13 9.66 26.56 1,79 2.03 18.53 7.6 41/71 7.39 29.19 4.89 27.24 6.14 28.22 1.77 1.38 28.79 4.8 64 5.44 13.39 3.99 11.86 4.71 12.63 1.03 1.08 21.77 8.3 40 3.22 12.09 0.00 0.00 1.61 6.05 2.28 8.55 100 0.72 1.17 0.00 0.72 0.36 0.95 0.51 0.32 33. 63 1.24 3.89 0.79 4.05 1.02 3.97 0.32 0.11 31.35 2.8 74 4.76 10.20 4.61 8.29 4.69 9.24 0.11 1.35 2.26 14. 70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18.			39.57		32.50	11.18	36.04	2.51	5.00	22.46	13.87
41/71 7.39 29.19 4.89 27.24 6.14 28.22 1.77 1.38 28.79 4.8 64 5.44 13.39 3.99 11.86 4.71 12.63 1.03 1.08 21.77 8.5 40 3.22 12.09 0.00 0.00 1.61 6.05 2.28 8.55 100 0.72 1.17 0.00 0.72 0.36 0.95 0.51 0.32 33. 63 1.24 3.89 0.79 4.05 1.02 3.97 0.32 0.11 31.35 2.8 74 4.76 10.20 4.61 8.29 4.69 9.24 0.11 1.35 2.26 14. 70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18. 66 20.48 42.53 15.66 38.23 18.07 40.38 3.41 3.04 18.86 7.3			28.00	8,39	25.13	9.66	26.56	1.79			7.64
64 5.44 13.39 3.99 11.86 4.71 12.63 1.03 1.08 21.77 8.5 40 3.22 12.09 0.00 0.00 1.61 6.05 2.28 8.55 100 0.72 1.17 0.00 0.72 0.36 0.95 0.51 0.32 33. 63 1.24 3.89 0.79 4.05 1.02 3.97 0.32 0.11 31.35 2.8 74 4.76 10.20 4.61 8.29 4.69 9.24 0.11 1.35 2.26 14. 70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18. 66 20.48 42.53 15.66 38.23 18.07 40.38 3.41 3.04 18.86 7.5 95 4.43 1.90 3.93 0.00 4.18 0.95 0.35 1.35 8.47 141 <th< td=""><td></td><td></td><td>29.19</td><td>4.89</td><td>27.24</td><td>6.14</td><td>28.22</td><td>1.77</td><td>1.38</td><td></td><td>4.89</td></th<>			29.19	4.89	27.24	6.14	28.22	1.77	1.38		4.89
40 3.22 12.09 0.00 0.00 1.61 6.05 2.28 8.55 100 0.72 1.17 0.00 0.72 0.36 0.95 0.51 0.32 33 63 1.24 3.89 0.79 4.05 1.02 3.97 0.32 0.11 31.35 2.8 74 4.76 10.20 4.61 8.29 4.69 9.24 0.11 1.35 2.26 14 70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18 66 20.48 42.53 15.66 38.23 18.07 40.38 3.41 3.04 18.86 7.5 95 4.43 1.90 3.93 0.00 4.18 0.95 0.35 1.35 8.47 141 91 0.54 1.79 0.34 0.68 0.44 1.23 0.14 0.78 32.14 63. 56/60			13.39	3.99	11.86	4.71	12.63	1.03			8.57
63 1.24 3.89 0.79 4.05 1.02 3.97 0.32 0.11 31.35 2.8 74 4.76 10.20 4.61 8.29 4.69 9.24 0.11 1.35 2.26 14. 70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18. 66 20.48 42.53 15.66 38.23 18.07 40.38 3.41 3.04 18.86 7.5 95 4.43 1.90 3.93 0.00 4.18 0.95 0.35 1.35 8.47 141 91 0.54 1.79 0.34 0.68 0.44 1.23 0.14 0.78 32.14 63. 56/60 9.67 18.18 7.06 14.67 8.37 16.42 1.85 2.48 22.06 15. 89 0.45 0.42 0.47 0.28 0.46 0.35 0.01 0.10					0.00	1.61	6.05	2.28			1
63 1.24 3.89 0.79 4.05 1.02 3.97 0.32 0.11 31.35 2.8 74 4.76 10.20 4.61 8.29 4.69 9.24 0.11 1.35 2.26 14. 70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18. 66 20.48 42.53 15.66 38.23 18.07 40.38 3.41 3.04 18.86 7.5 95 4.43 1.90 3.93 0.00 4.18 0.95 0.35 1.35 8.47 141 91 0.54 1.79 0.34 0.68 0.44 1.23 0.14 0.78 32.14 63. 56/60 9.67 18.18 7.06 14.67 8.37 16.42 1.85 2.48 22.06 15. 89 0.45 0.42 0.47 0.28 0.46 0.35 0.01 0.10			1.17	0.00	0.72	0.36	0.95	0.51			33.67
74 4.76 10.20 4.61 8.29 4.69 9.24 0.11 1.35 2.26 14. 70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18. 66 20.48 42.53 15.66 38.23 18.07 40.38 3.41 3.04 18.86 7.5 95 4.43 1.90 3.93 0.00 4.18 0.95 0.35 1.35 8.47 141 91 0.54 1.79 0.34 0.68 0.44 1.23 0.14 0.78 32.14 63. 56/60 9.67 18.18 7.06 14.67 8.37 16.42 1.85 2.48 22.06 15. 89 0.45 0.42 0.47 0.28 0.46 0.35 0.01 0.10 3.11 28. 92/84 4.59 8.20 4.47 5.36 4.53 6.78 0.08 2.01					4.05	1.02	3.97	0.32		31.35	2.85
70/76 21.68 30.13 15.35 23.28 18.51 26.70 4.48 4.84 24.18 18 66 20.48 42.53 15.66 38.23 18.07 40.38 3.41 3.04 18.86 7.5 95 4.43 1.90 3.93 0.00 4.18 0.95 0.35 1.35 8.47 141 91 0.54 1.79 0.34 0.68 0.44 1.23 0.14 0.78 32.14 63. 56/60 9.67 18.18 7.06 14.67 8.37 16.42 1.85 2.48 22.06 15. 89 0.45 0.42 0.47 0.28 0.46 0.35 0.01 0.10 3.11 28. 92/84 4.59 8.20 4.47 5.36 4.53 6.78 0.08 2.01 1.87 29. 101 6.71 2.61 5.78 0.46 6.24 1.53 0.66 1.52					8.29			0.11			14.61
66 20.48 42.53 15.66 38.23 18.07 40.38 3.41 3.04 18.86 7.5 95 4.43 1.90 3.93 0.00 4.18 0.95 0.35 1.35 8.47 141 91 0.54 1.79 0.34 0.68 0.44 1.23 0.14 0.78 32.14 63. 56/60 9.67 18.18 7.06 14.67 8.37 16.42 1.85 2.48 22.06 15. 89 0.45 0.42 0.47 0.28 0.46 0.35 0.01 0.10 3.11 28. 92/84 4.59 8.20 4.47 5.36 4.53 6.78 0.08 2.01 1.87 29. 101 6.71 2.61 5.78 0.46 6.24 1.53 0.66 1.52 10.53 99. 99 4.59 2.09 3.80 1.67 4.19 1.88 0.56 0.30 1						18.51	26.70	4.48			18.14
95 4.43 1.90 3.93 0.00 4.18 0.95 0.35 1.35 8.47 141 91 0.54 1.79 0.34 0.68 0.44 1.23 0.14 0.78 32.14 63. 56/60 9.67 18.18 7.06 14.67 8.37 16.42 1.85 2.48 22.06 15. 89 0.45 0.42 0.47 0.28 0.46 0.35 0.01 0.10 3.11 28. 92/84 4.59 8.20 4.47 5.36 4.53 6.78 0.08 2.01 1.87 29. 101 6.71 2.61 5.78 0.46 6.24 1.53 0.66 1.52 10.53 99. 99 4.59 2.09 3.80 1.67 4.19 1.88 0.56 0.30 13.32 15. 119 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.28 0.68 </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td>40.38</td> <td>3.41</td> <td></td> <td></td> <td>7.53</td>							40.38	3.41			7.53
91 0.54 1.79 0.34 0.68 0.44 1.23 0.14 0.78 32.14 63. 56/60 9.67 18.18 7.06 14.67 8.37 16.42 1.85 2.48 22.06 15. 89 0.45 0.42 0.47 0.28 0.46 0.35 0.01 0.10 3.11 28. 92/84 4.59 8.20 4.47 5.36 4.53 6.78 0.08 2.01 1.87 29. 101 6.71 2.61 5.78 0.46 6.24 1.53 0.66 1.52 10.53 99. 99 4.59 2.09 3.80 1.67 4.19 1.88 0.56 0.30 13.32 15. 119 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.28 4.35 34. 97 2.24 0.76 1.93 0.60 2.08 </td <td></td> <td></td> <td></td> <td></td> <td></td> <td>4.18</td> <td>0.95</td> <td>0.35</td> <td></td> <td></td> <td>141.42</td>						4.18	0.95	0.35			141.42
56/60 9.67 18.18 7.06 14.67 8.37 16.42 1.85 2.48 22.06 15. 89 0.45 0.42 0.47 0.28 0.46 0.35 0.01 0.10 3.11 28. 92/84 4.59 8.20 4.47 5.36 4.53 6.78 0.08 2.01 1.87 29. 101 6.71 2.61 5.78 0.46 6.24 1.53 0.66 1.52 10.53 99. 99 4.59 2.09 3.80 1.67 4.19 1.88 0.56 0.30 13.32 15. 119 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.28 4.35 34. 97 2.24 0.76 1.93 0.60 2.08 0.68 0.22 0.11 10.54 16.				0.34	0.68	0.44					63.81
89 0.45 0.42 0.47 0.28 0.46 0.35 0.01 0.10 3.11 28. 92/84 4.59 8.20 4.47 5.36 4.53 6.78 0.08 2.01 1.87 29. 101 6.71 2.61 5.78 0.46 6.24 1.53 0.66 1.52 10.53 99. 99 4.59 2.09 3.80 1.67 4.19 1.88 0.56 0.30 13.32 15. 119 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 83 0.67 1.03 0.63 0.63 0.65 0.83 0.03 0.28 4.35 34. 97 2.24 0.76 1.93 0.60 2.08 0.68 0.22 0.11 10.54 16.			18.18		14.67	8.37					15.12
92/84 4.59 8.20 4.47 5.36 4.53 6.78 0.08 2.01 1.87 29. 101 6.71 2.61 5.78 0.46 6.24 1.53 0.66 1.52 10.53 99. 99 4.59 2.09 3.80 1.67 4.19 1.88 0.56 0.30 13.32 15. 119 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 83 0.67 1.03 0.63 0.63 0.65 0.83 0.03 0.28 4.35 34. 97 2.24 0.76 1.93 0.60 2.08 0.68 0.22 0.11 10.54 16.	89			0.47	0.28						28.08
101 6.71 2.61 5.78 0.46 6.24 1.53 0.66 1.52 10.53 99. 99 4.59 2.09 3.80 1.67 4.19 1.88 0.56 0.30 13.32 15. 119 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 83 0.67 1.03 0.63 0.63 0.65 0.83 0.03 0.28 4.35 34. 97 2.24 0.76 1.93 0.60 2.08 0.68 0.22 0.11 10.54 16.	92/84	4.59	8.20	4.47	5.36	4.53					29.62
99 4.59 2.09 3.80 1.67 4.19 1.88 0.56 0.30 13.32 15. 119 0.00<		6.71	2.61	5.78							99.20
119 0.00	99	4.59	2.09	3.80							15.78
83 0.67 1.03 0.63 0.63 0.65 0.83 0.03 0.28 4.35 34. 97 2.24 0.76 1.93 0.60 2.08 0.68 0.22 0.11 10.54 16.	119	0.00	0.00	0.00						- /	
97 2.24 0.76 1.93 0.60 2.08 0.68 0.22 0.11 10.54 16.	83	0.67	1.03							4,35	34.28
10.01	97										16.58
	87	3.50	0.96	3.12	0.45	3.31	0.70	0.27	0.36	8.13	51.33
											1.32

Table 4. (continued) Precision expressed as RSD for congeners and total PCB based on measurement of duplicate samples from site B12 on June 27, 1996. Units are in pg/L.

Congener	SCXX	0627	DCXX	627	Average 1	Dup	Variation	(SD)	%RSD	%RSD
BZ#	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.
110/77	13.39	6.85	10.86	5.15	12.12	6.00	1.79	1.20	14.76	20.03
82	1.29	2.54	1.15	1.71	1.22	2.12	0.10	0.59	8.13	27.68
151	1.70	1.13	1.53	0.46	1.61	0.79	0.12	0.47	7.45	59.78
107	0.00	0.00	0.00	0.94	0.00	0.47	0.00	0.66		
149	5.30	1.77	4.33	1.03	4.82	1.40	0.69	0.52	14.24	37.31
118	5.70	3.23	4.24	1.79	4.97	2.51	1.03	1.02	20.77	40.65
134/114	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
131	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
146	1.59	0.30	1.49	0.74	1.54	0.52	0.07	0.31	4.59	60.12
153/132/105	19.74	5.46	15.78	5.27	17.76	5.36	2.80	0.13	15.76	2.51
141	3.05	0.80	2.69	0.58	2.87	0.69	0.25	0.16	8.87	40.43
137/176	13.01	9.38	8.49	6.22	10.75	7.80	3.20	2.23	29.73	28.65
130	0.00	0.00	0.00	0.23	0.00	0.12	0.00	0.16		
138/163	12.03	3.03	9.16	2.09	10.59	2.56	2.03	0.66	19.16	25.99
158	1.77	1.42	1.23	1.37	1.50	1,39	0.38	0.04	25.46	2.54
129	0.97	0.00	0.58	0.00	0.78	0.00	0.28	0.00	35.58	
178	0.64	0.80	0.40	0.00	0.52	0.40	0.17	0.57	32.64	
187/182	3.29	0.91	2.77	0.02	3.03	0.46	0.37	0.63	12.16	136.07
183	1.71	0.45	1.58	0.00	1.65	0.23	0.09	0.32	5.59	
128	1.78	0.00	1.44	0.00	1.61	0.00	0.24	0.00	14.93	
167	0.50	0.82	0.00	0.70	0.25	0.76	0.35	0.08		11.16
185	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.01	~~~
174	1.54	0.23	1.58	0.04	1.56	0.14	0.03	0.13	1.81	97.71
177	1.86	0.00	1.55	0.70	1.71	0.35	0.22	0.49	12.86	
173	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
157/200	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.54	
180	5.09 0.00	0.00	4.98 0.00	0.00	5.04	0.00	0.08	0.00	1.54	
193 191		0.00		0.00	0.00	0.00	0.00	0.00		
191	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
170/190	5.57	1.04	4.57	0.43	5.07	0.00	0.00	0.00	13.95	58.89
198	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	13.55	30,03
201	2.61	0.85	3.45	0.00	3.03	0.43	0.59	0.60	19.62	
203/196	2.86	1.39	4.53	0.00	3.69	0.69	1.18	0.98	32.00	
189	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	52.00	
208/195	0.00	0.00	2.51	0.00	1.26	0.00	1.77	0.00		
207	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
194	0.65	0.32	1.64	0.00	1.14	0.16	0.70	0.22	61.41	
205	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	01.11	
206	1.38	0.39	2.16	0.00	1.77	0.20	0.55	0.38	31.20	
209	0.81	0.00	1.09	0.15	0.00	0.00		1	21,20	
	0.01		1107		0,00	1 0.00		<u> </u>	Ave Cong	ener RSD
								Mean	18.85	26.53
			Total	DCD				S.D.	11.15	26.95
			<u>Total</u>	rub	•			S.D.	11.13	40.73

Total	PCB
потаг	PUB

Total PCB	Sexx	0627	Dexx	0627	Average	Dup	Variation	(S.D.)	Total PCB I	RSD (%)
	Part.	Diss.	Part.		Part.	Diss.	Part.	Diss.	Part.	Diss.
pg/L	372.3	853.0	287.1	742.7	328.8	797.8	60.3	78.0	18.33	9.78
ng/L		1.23		1.03		1.13		0.14]	12.27

Table 5. Precision expressed as RSD for congeners and total PCB based on measurement of duplicate samples from site B12 on August 20, 1996. Units are in pg/L.

								ire in pg/L		
Congener		X0820		XX0627		ge Dup		Variation	%RSD	%RSD
BZ#	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.
11	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
3	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
10/4	0.00		0.00		0.00		0.00	<u> </u>		
7	0.00	0.00	0.00	0.00	0,00	0.00	0.00	0.00		
6	0.00	4.85	0.00	4.22	0.00	4.54	0.00	0.45		9.82
19	0.00	3.48	0.00	0.00	0.00	1.74	0.00	2.46		
18	2.32	34.58	2.54	30.83	2.43	32.71	0.16	2.65	6.40	8.11
17	0.87	22.69	1.03	19.17	0.95	20.93	0.11	2.49	11.91	11.89
27	0.00	2.32	0.00	2.09	0.00	2.21	0.00	0.16		7.38
16/32	0.00	26.42	1.04	23.47	0.52	24.95	0.74	2.09		8.36
29	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
26	0.00	11.76	0.00	. 9.50	0.00	10.63	0.00	1.60		15.03
25	0.00	4.72	0.00	4.51	0.00	4.62	0.00	0.15		3,22
31/28	7.84	88.22	8.35	77.48	8.10	82.85	0.36	7.59	4.45	9.17
33	0.00	8.99	0.00	6.59	0.00	7.79	0.00	1.70		21.79
53	0.00	4.80	0.00	4.49	0.00	4.65	0.00	0.22		4.72
51	0.00	0.54	0.00	0.42	0.00	0.48	0.00	0.08		17.68
22	0.00	11.99	0.00	9.37	0.00	10.68	0.00	1.85		17.35
45	0.00	3.28	0.00	2.98	0.00	3.13	0.00	0.21		6.78
46	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
52	3.20	9.44	3.47	6.87	3.34	8.16	0.19	1.82	5.72	22.28
49	2.13	12.13	2.45	9.95	2.29	11.04	0.23	1.54	9.88	13.96
47	2.12	1.63	2.73	5.33	2.43	3.48	0.43	2.62	17.79	75.18
48	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
44	3.16	13.96	2.87	11.70	3.02	12.83	0.21	1.60	6.80	12.46
37/42	1.32	12.21	1.30	11.13	1.31	11.67	0.01	0.76	1.08	6.54
41/71	0.00	12.49	0.00	11.57	0.00	12.03	0.00	0.65		
64	0.92	5.77	1.28	5.37	1.10	5.57	0.25	0.28	23.14	5.08
40	0.00	0.00	0.00	8.54	0.00	4.27	0.00	6.04		
100	0.00	0.00	0.00	0.38	0.00	0.19	0.00	0.27		
63	0.00	1.06	0.00	0.00	0.00	0.53	0.00	0.75		
74	1.77	3.18	2.02	1.90	1.90	2.54	0.18	0.91	9.33	35.63
70/76	3.95	9.65	4.27	8.15	4.11	8.90	0.23	1.06	5.51	11.92
66	0.00	19.49	0.00	15.29	0.00	17.39	0.00	2.97		17.08
95	2.06		2.21		2.14	ł !	0.11]	4.97	
91	0.06	į	0.06		0.06		00.00		0.00	
56/60	1.71	5.32	1.41	4.35	1.56	4.84	0.21	0.69	13.60	14.19
89	0.16		0.19		0.18	i i	0.02		12.12	
92/84	1.48	1.94	1.69	1.77	1.59	1.86	0.15	0.12	9.37	6.48
101	1.78	<u> </u>	1.82		1.80	<u> </u>	0.03	<u>i</u>	1.57	
99	0.68		1.61		1.15	<u> </u>	0.66	<u> </u>	57.43	
119	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
83	0.00		0.29		0.15		0.21		<u> </u>	
97	0.62		0.63		0.63		0.01		1.13	
87	1.32		1,15		1.24		0.12		9.73	
85	2.44	3.88	2.84	3,85	2.64	3.87	0.28	0.02	10.71	0.55
136	0.27		0.31		0.29		0.03		9.75	
110/77	3.83		3.79		3.81		0.03		0.74	
82	0.56	0.24	0.46		0.51	0.24	0.07		13.86	
151	0.50		0.55		0.53		0.04		6.73	
107	0.00	0.00	0.00	0.69	0.00	0.35	0.00	0.49		
149	1.44		1.48		1.46		0.03	[1.94	

Table 5. (continued). Precision as RSD for congeners and total PCB based on measurement of duplicate samples from site B12 on August 20, 1996. Units are in pg/L.

Congener	SCXX		511100000000000000000000000000000000000	X0627	Average		Variatio		%RSD	%RSD
BZ#	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.
118	1.24	0.37	1,29		1.27	0.37	0.04		2.79	
134/114	0.00		0.00		0.00	0.00	0.00			
131	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
146	0.60		0.70		0.65		0.07		10.88	
153/132/105	6.67		5.13		5.90		1.09		18.46	
141	0.79		1.08		0.94		0,21		21.93	
137/176	0.00		0.00		0.00		0.00			
130	0.00	0.17	0.00	0.00	0.00	0.09	0.00	0.12		
138/163	2.54		2.80		2.67		0.18		6.89	
158	0.00	0.04	0.00		0.00	0.04	0.00			
129	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
178	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
187/182	0.86	0.41	0.89		0.88	0.41	0.02	<u> </u>	2.42	
183	0.00	0.16	0.54		0.27	0.16	0.38			
128	0.75		0.68		0.72	0.00	0.05		6.92	
167	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
185	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
174	0.56	0.37	0.60		0.58	0.37	0.03	ļ	4.88	
177	0.58	0.55	0.59	0.00	0.59	0.28	0.01	0.39	1.21	
173	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	_	
157/200	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
180	0.86	1	0.73	<u> </u>	0.80	0.00	0.09	<u> </u>	11.56	
193	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
191	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	<u> </u>	
199	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	<u> </u>	
170/190	1.00	0.33	1.25	0.00	1.13	0.33	0.18	0.00	15.71	
198	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1 200	
201	0.48	1.75	0.50	ļ	0.49	1.75	0.01	 	2.89	
203/196	0.63	2.24	0.35	0.00	0.49	2.24	0.20	0.00	40.41	
189	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	 	
208/195	0.00	0.94	0.00	0.00	0.00	0.00	0.00	1 000		
207	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	-	
194	0.00	0.77	0.00	0.00	0.00	0.77	0.00	0.00	1	
205	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	i	
206		0.68		 	0.00			<u> </u>	1	
209		0.00		!	0.00	0.00	<u> </u>	<u> </u>		

Total PCB

	Ave. Congener RSD							
Mean	10.32	14.51						
S.D.	10.98	14.66						

Total PCB	Sexx 0627		Dexx 0627		Dup.		Variation (S.D.)		Total PCB RSD (%)	
	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.	Part.	Diss.
pg/L	66.07	349.81	70.97	301.96	68.52	329.10	3.46	33.84	5.06	10.28
ng/L	0.416		0.373		0.398		0.030		7.639	

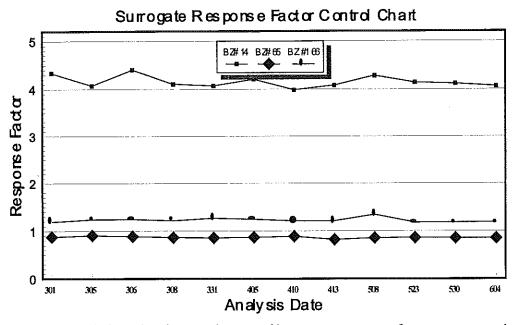


Figure 5. Control chart showing consistency of instrument response for surrogate standards.

Accuracy

In the absence of a valid quality control check sample for low-level PCB analysis in real water samples, we used a combination of matrix spike recovery and surrogate spike recovery procedures. In surrogate spike recovery, every sample is spiked with a mixture of three congeners (14, 65, 166). The original spiking solution is run as a standard to quantify recovery, including internal standard correction with congeners 30 and 204. This procedure verifies the integrity of the extraction/concentration procedure for every sample, however it does not verify the validity of the standard solution (610 performance check standard) and data reduction procedure.

In matrix spike recovery, selected duplicate water samples are spiked with 1ml of a dilution of a PCB calibration check solution (C-CCSEC from Accustandards), which contains 20 congeners (8,18,28,44,52,66,77,101,105,118,126,128,138,153,170,180,187,195,206,209) ranging from dichloro- to decachlorobiphenyls. Two of these congeners (153/105) coelute on most DB-5 columns and are quantified together (Swackhamer, 1988). In this investigation we are not

quantifying congeners 8/5 since they are interfered with by αBHC and congener 126 is also not quantified. This leaves spike recovery determinations on 18 congeners represented by 17 peaks. In some cases, two congeners are identified together (e.g. 110/77) although only one was spiked (e.g. 77).

The spiked sample is quantified using the 610 performance standard and is taken through the same data reduction program of congener identification/quantification as each sample. As a result, the matrix spike recovery procedure serves as a true check on both the 610 performance standard and the data reduction program and should thus truly document the overall accuracy of the procedure.

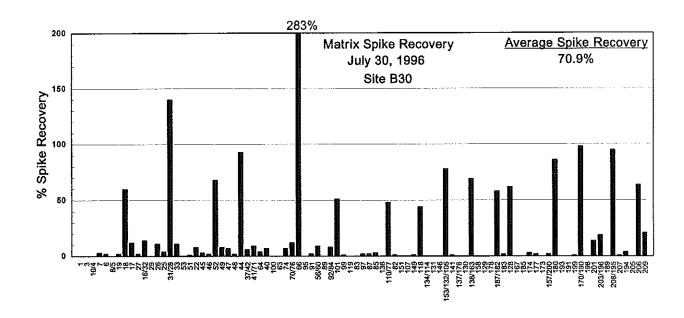
Matrix Spike Recovery

Results for two matrix spike analyses are shown for two samples (B30 and B45) taken during the middle and last sample run of Lake Champlain in Figure 5. Overall average spike recoveries for the 17 congeners were $70.9 \pm 26.7\%$ and $64.5 \pm 27.1\%$ for the two samples, respectively. Matrix spike recoveries of this magnitude and variability are typical of most complex organic extraction/concentration procedures (e.g. Swackhamer, 1988).

Several poor spike recoveries are noteworthy. In both cases, congener 209 showed very poor spike recovery (22 and 21%). This may be due to inaccurate mass assignment in the 610 performance standard (Swackhamer, 1988) or inefficient extraction. If the latter is the case, it likely was not adequately extracted off the hydrophobic XAD resin by the acetone/hexane soxhlet mixture. In one case, congener 66 showed inordinately high recoveries (283% and 135%), possibly due to misidentification of a contaminant as congener 166. The 283% recovery was not used in the calculation of the mean recovery since it would have biased the apparent recovery from 71% up to 85%. Finally, congener 77 was not identified in the B45 sample (Fig. 5). This congener is quantified as a coeluting congener with 110. When congeners of similar mass coelute as a single peak, the retention time is modified to a compromise between the two. Using a matrix spike of only one of those compounds (not representative of the sample or the 610 standard) probably shifted the retention time outside of the retention window.

Counting these problems with congeners 66, 77 and 209, matrix spike recovery was adequate for the purposes of this investigation in terms of congener identification and quantification for 30 of the 34 congeners quantified. Furthermore, examination of Fig. 5 shows a consistent pattern of matrix spike recovery across the two samples.

In the Green Bay Mass Balance Study (Swackhamer, 1988) matrix spiking was done with the performance standard, which was also used to quantify spike recovery. Recoveries between 50 and 120% were considered adequate. However, in the present study, spiking was done with a totally independent standard, thus the matrix spike recovery procedure was a true worst-case scenario for overall quantitation.



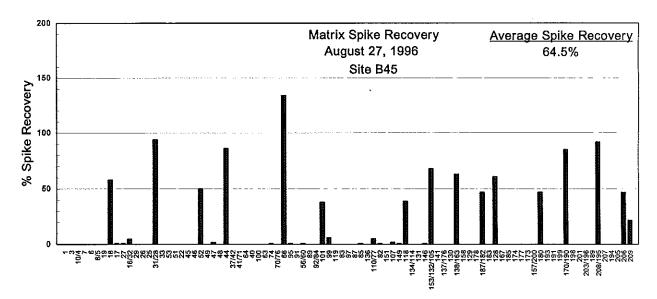


Figure 6. Matrix spike recovery for two samples from site B30 and B45 spiked with one ml of a PCB calibration check solution (Accustandard) containing 20 congeners (17 quantified) at a concentration of 10 ng/ml.

Surrogate Spike Recovery

Table 3 shows results for surrogate spike recovery for lake samples and QA samples in both the dissolved and particulate phases. Average spike recoveries in the particulate phase were 94±5%, 103±5% and 102±6% for congeners 14, 65 and 166, respectively. Average spike recoveries for the dissolved phase were 100±12%, 108±8% and 81±6% for the same congeners. Note that spike recovery was poorest for the most hydrophobic congener (166) in the dissolved phase, which is also captured on the XAD resin. This would support the hypothesis above that matrix spike recovery of congener 209 is poor due to loss on the XAD resin. Note that although the surrogate spike recoveries are better than the matrix spike recoveries, they do not verify all of the steps in the procedure.

Table 6. Surrogate spike recoveries (%) from samples analyzed in this study.

				Dissolved	Particulate			
Sample	Site	Date	BZ#14	BZ#65	BZ#166	BZ#14	BZ#65	BZ#166
SAXR0416	Saranac River	April 16	85.4	89.1	71.1	104.7	109.8	112.0
SAXR0418	Saranac River	April 18	108.6	103.4	63.4	96.2	111.1	98.5
SGXR0612	Stave Island	June 12	114.7	121.2	85.8	105.6	111.7	108.1
SFXR0614	B45	June 14	111.2	108.9	77.2	104.2	110.7	108.4
SHXR0618	The Gut	June 18	109.3	117.3	83.8	96.7	106.4	104.5
SBXR0625	B5	June 25	108.5	117.7	90.5	96.5	1.801	111.4
SEXR0626	B30	June 26	104.0	117.2	82.8	96.3	108.1	109.2
SCXR0627	B12	June 27	111.8	123.9	97.5	92.8	107.5	111.0
DCXR0627	Duplicate	June 27	89.3	102.9	81.5	93.5	100.5	105.7
SFXR0703	B45	July 3	99.8	109.5	84.9	90.2	100.7	101.5
SGXR0709	Stave Island	July 9	100.6	112.6	84.8	87.7	98.9	99.2
SEXR0711	B30	July 11	65.3	106.4	84.3	89.6	101.3	98.4
SDXR0715	B15	July 15	104.8	114.9	8.18	90.9	99.3	99.4
SBXR0716	B5	July 16	110.7	104.1	83.1	93.1	104.7	101.7
SFXR0724	B45	July 24	106.4	112.8	78.2	90.5	97.2	97.4
SGXR0725	Stave Island	July 25	102.6	109.9	80.2	91.4	104.5	95.6
SIXR0729	Four Brothers	July 29	109.2	112.5	82.6	88.5	101.8	93.6
SEXR0730	B30	July 30	69.2	99.0	75.7	91.3	100.9	97.2
SDXR0812	B15	Aug. 12	107.8	108.3	77.4	90.6	97.6	95.6
SBXR0813	B5	Aug. 13	102.0	101.3	79.4	97.5	100.3	100.6
SHXR0819	The Gut	Aug. 19	90.5	101.3	84.1	94.4	101.4	103.5
SCXR0820	B12	Aug. 20	98.0	101.8	80.5	90.9	100.5	100.5
DCXR0820	Duplicate	Aug. 20	95.1	99.2	86.8	96.4	98.7	103.5
SIXR0826	Four Brothers		95.6	98.4	76.0	92.7	102.2	94.4
SFXR0827	B45	Aug. 27	94.5	98.8	77.5	91.9	101.1	93.5
L		Mean	99.8	107.7	81.2	94.2	103.4	101.8
Standard Error Standard Dev. Range			2.5	1.7	1.3	1.0	0.9	1.1
			12.4	8.4	6.4	4.9	4.5	5.7
			49.3	34.8	34.1	17.8	14.6	18.5
	<u> </u>	Minimum	65.3	89.1	63.4	87.7	97.2	93.5
	<u></u>	Maximum	114.7	123.9	97.5	105.6	111.7	112.0

PCB Congener Results

Figures 7-16 on the following pages show congener patterns for water samples from Cumberland Bay, the Main Lake of Lake Champlain, and from the Saranac River. Lake results are summarized in figure 7 on the following page (see Table 2 for sample locations) and show a gradient in concentration from inner to outer Cumberland Bay and the adjacent Main Lake reference site (B45). Main Lake concentrations also suggest a North-South gradient, which would be consistent with the net northerly flow of Lake Champlain.

In most cases, coeluting congeners 31/28 were the major contributors to total PCBs in lake samples, often accounting for as much as 25-30%% of total PCB. This congener pair is also the most important representative in Aroclor 1242, although it only accounts for 11.1% of this Aroclor. This congener pair occurs as a peak doublet in Aroclor 1242 chromatograms with approximately 50% resolution and roughly equal peak heights. The possibility of a major contaminant being confused with 31/28 is doubtful, since any contaminant would enrich only one of the peaks. Examination of original lake sample chromatograms showed the 31 to 28 peak height ratio to be approximately the same as in Aroclor 1242. Apparent enrichment of this congener pair relative to a 1242 source could be due to fractionation of PCBs in the environment, with lighter congeners being volatilized or metabolized, and heavier congeners partitioning into sediments.

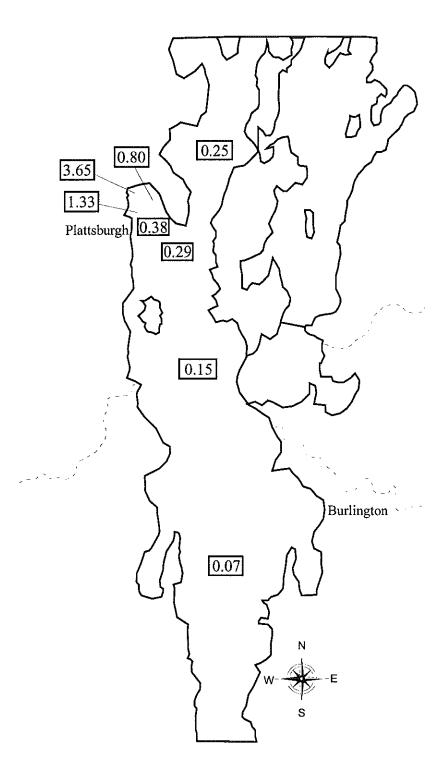


Figure 7. Distribution of average total PCBs (ng/L) in Cumberland Bay and the main lake portion of Lake Champlain in the summer of 1996.

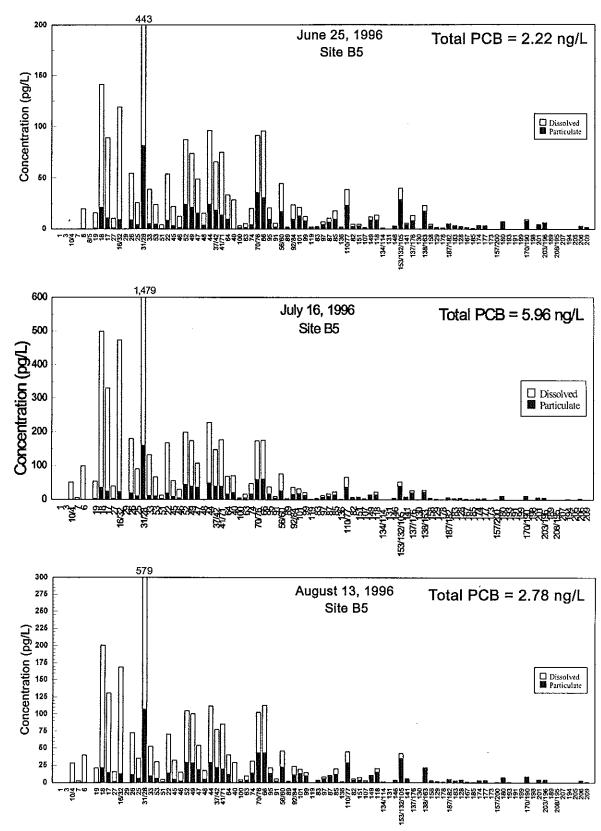


Figure 8. PCB congener distribution at site B5 in inner Cumberland Bay, Lake Champlain. Congener concentration less than the standard deviation of the blanks.

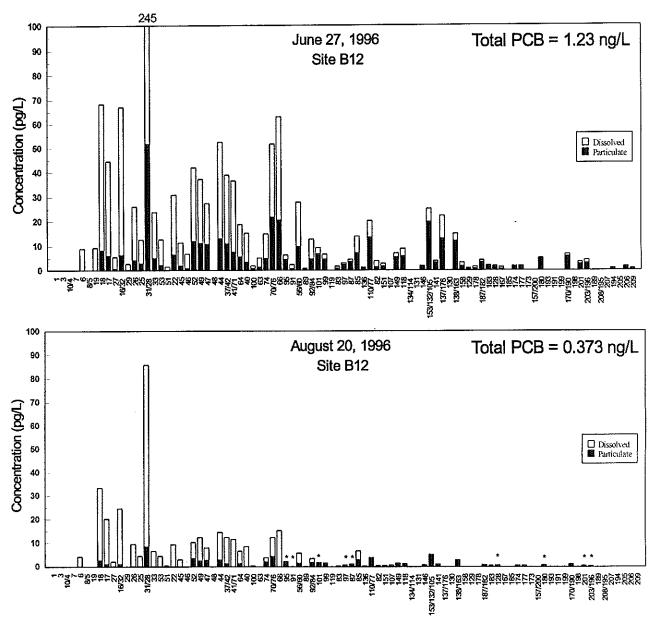


Figure 9. PCB congener distribution at site B12 in inner Cumberland Bay, Lake Champlain. *Congener concentration less than the standard deviation of the blanks.

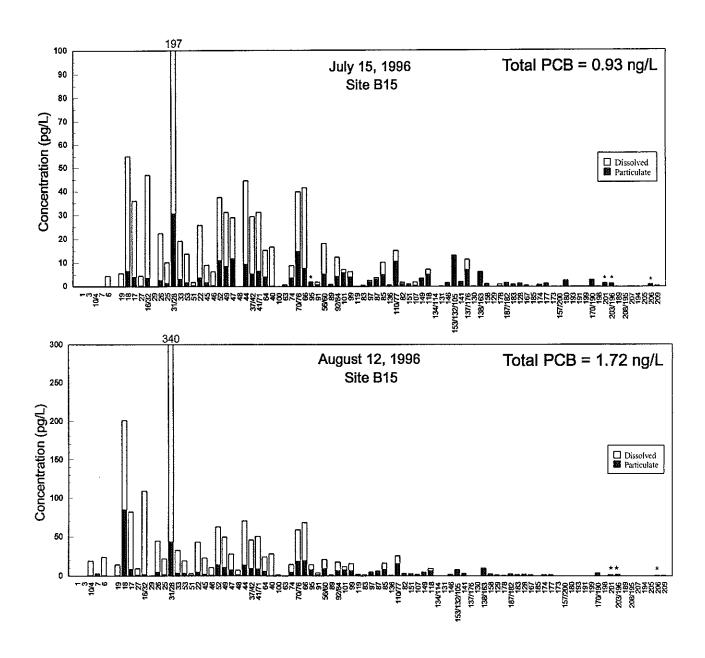


Figure 10. PCB congener distribution at site B15 in inner Cumberland Bay, Lake Champlain. *Congener concentration less than the standard deviation of the blanks.

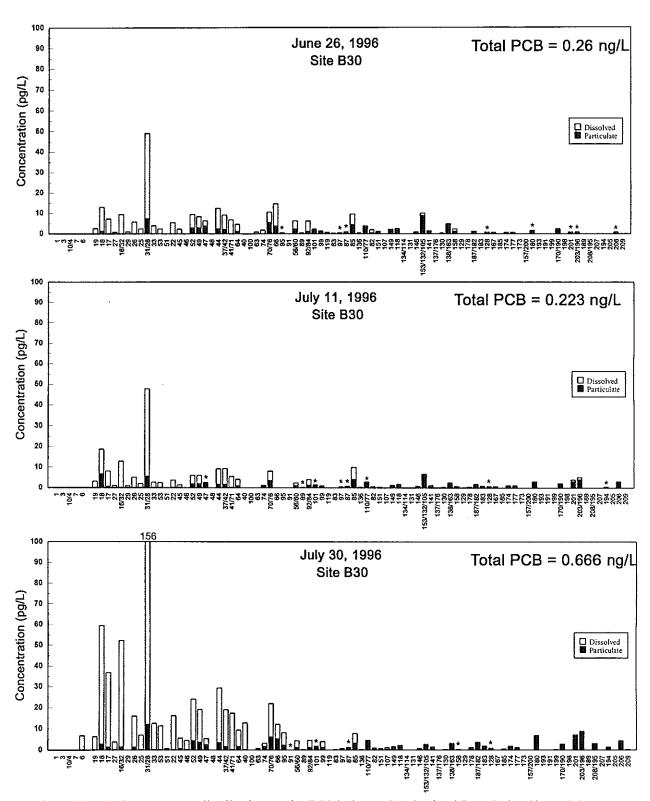


Figure 11. PCB congener distribution at site B30 in inner Cumberland Bay, Lake Champlain. *Congener concentration less than the standard deviation of the blanks.

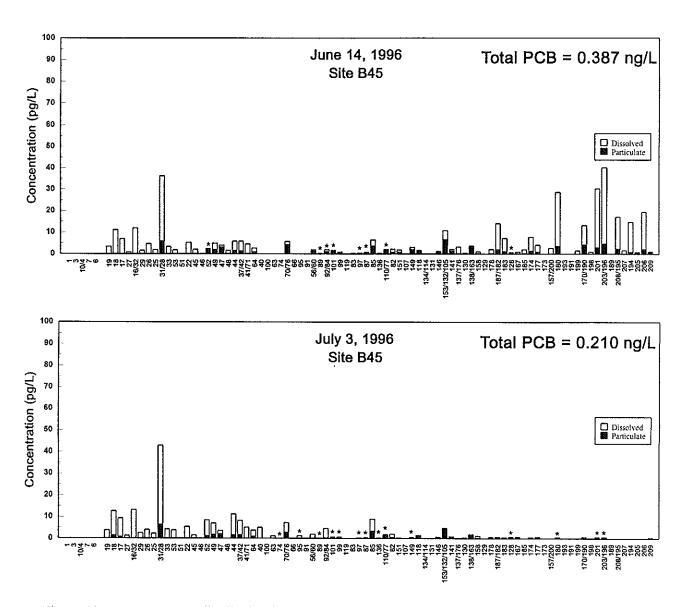


Figure 12. PCB congener distribution in two of the four samples from site B45 in the Main Lake east of Cumberland Bay, Lake Champlain. *Congener concentration less than the standard deviation of the blanks.

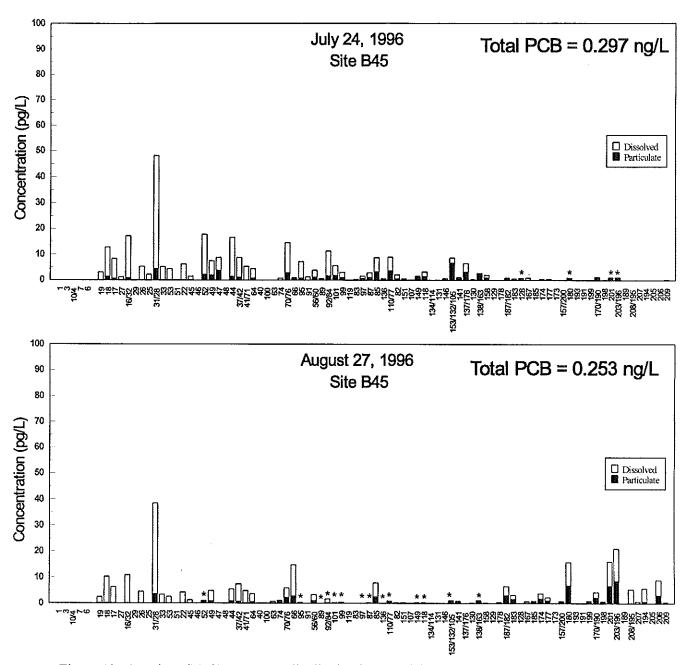


Figure 12. (continued) PCB congener distribution in two of the four samples from site B45 in the Main Lake east of Cumberland Bay, Lake Champlain. *Congener concentration less than the standard deviation of the blanks.

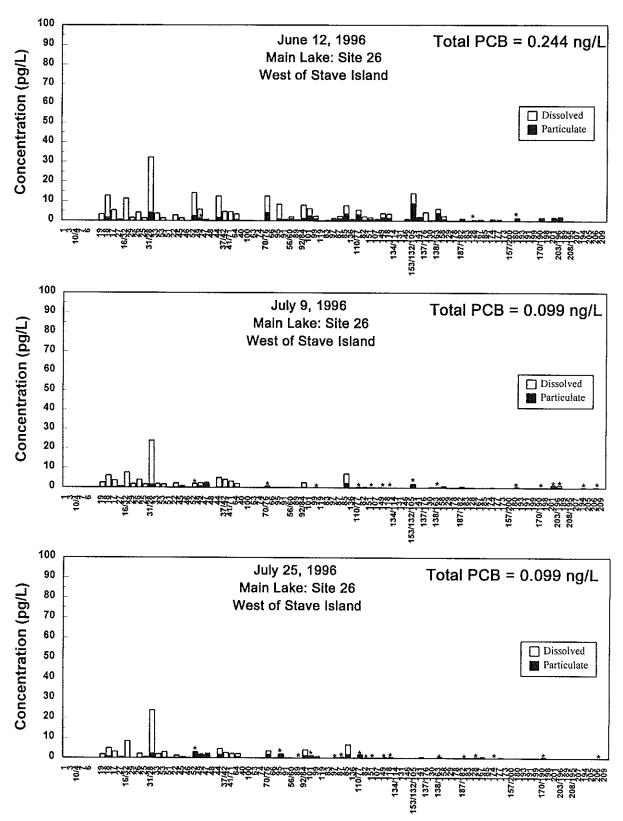
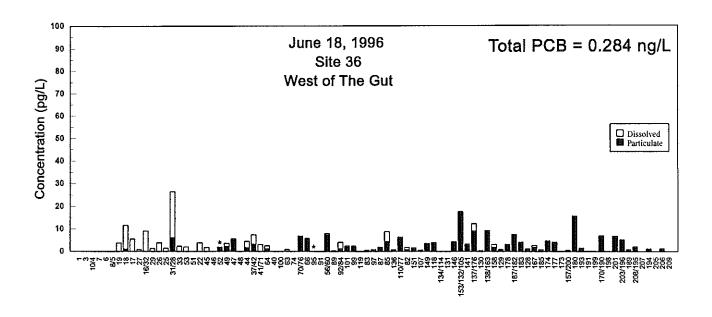


Figure 13. PCB congener distribution at site 26 in the Main Lake of Lake Champlain west of Stave Island. *Congener concentration less than the standard deviation of the blanks.



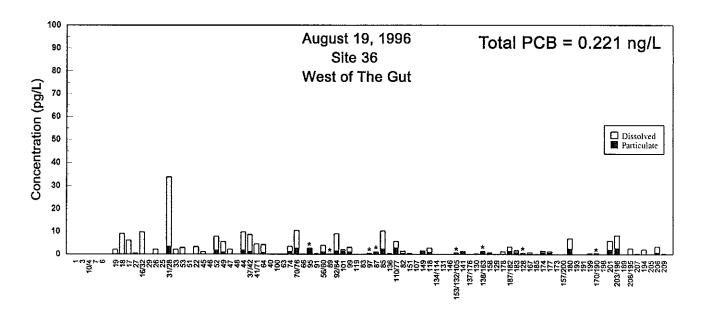
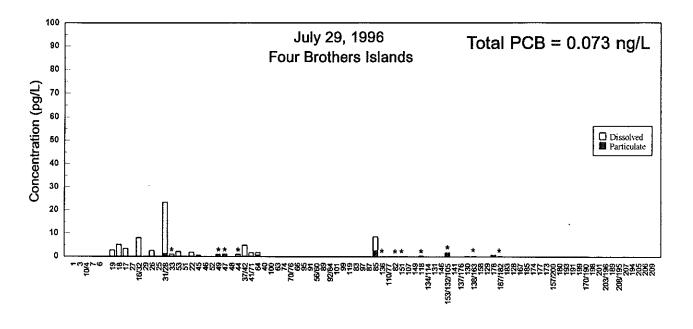


Figure 14. PCB congener distribution at site 36 in the Main Lake of Lake Champlain west of "The Gut". *Congener concentration less than the standard deviation of the blanks.



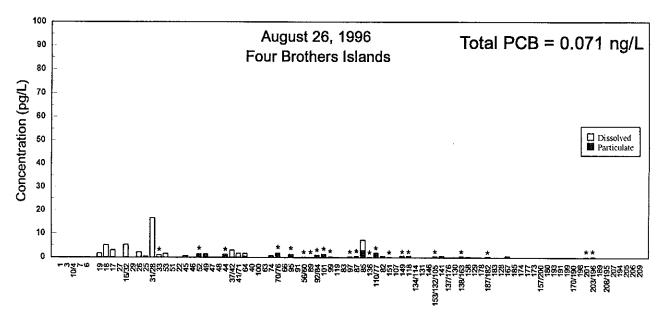


Figure 15. PCB congener distribution at site 14 in the Main Lake of Lake Champlain near South of Four Brothers Islands. *Congener concentration less than the standard deviation of the blanks.

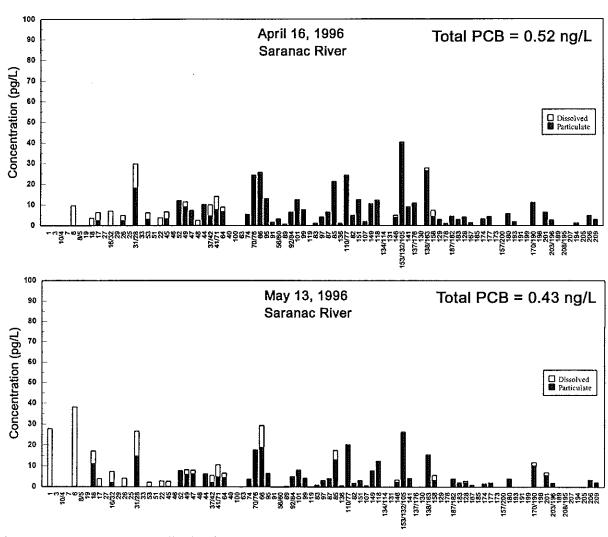


Figure 16. PCB congener distribution at the Bridge St. site on the Saranac River in Plattsburgh.

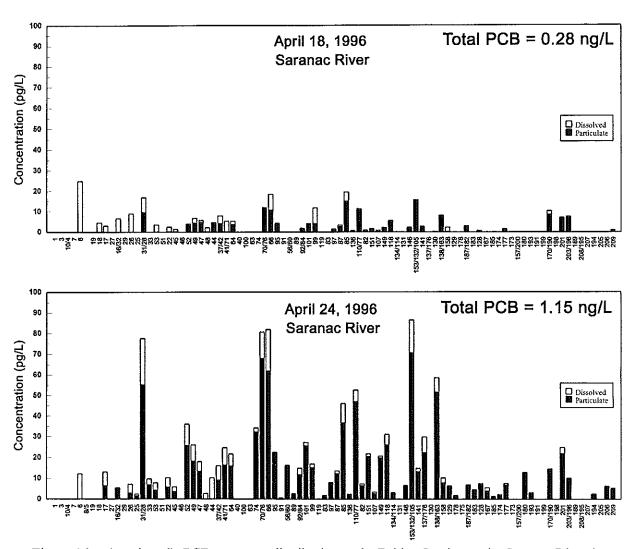


Figure 16. (continued) PCB congener distribution at the Bridge St. site on the Saranac River in Plattsburgh.

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