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Nonconstant Polychlorinated Biphenyl Partitioning in New Bedford Harbor Sediment during Sequential Batch Leaching

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■ Leaching of PCB-contaminated sediment from the New Bedford Harbor Superfund Site, New Bedford, MA, was conducted to assess the long-term mobilization of PCB. Sequential batch extraction of the sediment with distilled–deionized water resulted in a progressive increase in concentrations of PCB, dissolved organic carbon, and numbers of microorganisms in the extract. Sequential extraction with saline water produced extractant PCB concentrations that were relatively constant and significantly lower than those obtained with distilled–deionized water. The PCB mobilization pattern in distilled–deionized water extractant demonstrated dependence of PCB partitioning on ionic strength (as measured by conductivity). The dependency on ionic strength was related to release of sediment organic carbon as ionic strength decreased during sequential batch extraction. The data show that organic colloids and microparticulates mobilized by changes in ionic strength significantly facilitate the release of PCBs.

Introduction

Contaminant mobilization is a concern during removal (dredging) and disposal of sediments for cleanup of contaminated sites and maintenance of navigation channels. Contaminant mobility in dredged material is difficult to predict because of the complex nature of dredged material and because physicochemical conditions such as oxidation–reduction (redox) potential and pH at the disposal site affect contaminant mobility (*1*, *2*).

Polychlorinated biphenyls (PCBs) are members of an important class of chemicals, hydrophobic organic compounds, that are often present in contaminated sediments. Because PCBs do not have redox-dependent speciation and do not undergo significant acid/base dependent equilibria, the processes governing mobility are less difficult to describe and mathematically model than those of many other contaminants. Equilibrium partitioning is generally viewed as the proper basis for modeling the transport and fate of PCBs (*3*). In its simplest form, partitioning or distribution of a hydrophobic organic compound between sediment solids and water at equilibrium is mathematically written as follows (*4*):

$$K_d = C_s/C_w \qquad (1)$$

where C_s is the concentration of contaminant sorbed to the sediment solids (mg/kg), C_w the concentration of contaminant in the water phase (mg/L), and K_d the distribution coefficient (L/kg).

Theoretically, K_d is not time variable and depends only on sediment properties and the hydrophobic organic chemical of interest. Hydrophobic partitioning studies (*5–7*) have shown that K_d is largely determined by the organic content of the sediment. Other studies have shown that in batch experiments K_d is not constant, but may depend on the mass ratio of sediment solids to water (*8–16*). Because the equilibrium assumption implicit in all such studies is often difficult to fully justify, there is some controversy about the solids concentration dependency of distribution coefficients, but that issue is not the subject of this paper.

Table I. Total Sediment Concentration [mg/kg Dry Weight (Standard Error)] of PCBs (Triplicate Analyses) in New Bedford Sediment (Dry Weight Basis)

IUPAC[a] no.	compound	sediment concn
C7	2,4-dichlorobiphenyl	0.56 (0.01)
C8	2,4′-dichlorobiphenyl	166 (3.79)
C28	2,4,4′-trichlorobiphenyl	153 (5.2)
C44	2,2′,3,5′-tetrachlorobiphenyl	84.1 (3.52)
C49	2,2′,4,5′-tetrachlorobiphenyl	8.0 (0.85)
C50	2,2′,4,6-tetrachlorobiphenyl	153 (5.29)
C52	2,2′,5,5′-tetrachlorobiphenyl	177 (9.29)
C70	2,3′,4′,5-tetrachlorobiphenyl	59.2 (3.29)
C77	3,3′,4,4′-tetrachlorobiphenyl	147 (3.36)
C82	2,2′,3,3′,4-pentachlorobiphenyl	24.3 (1.21)
C87	2,2′,3,4,5′-pentachlorobiphenyl	8.2 (0.41)
C97	2,2′,3′,4,5-pentachlorobiphenyl	22.9 (1.13)
C101	2,2′,4,5,5′-pentachlorobiphenyl	70.4 (4.29)
C105	2,3,3′,4,4′-pentachlorobiphenyl	36.7 (0.88)
C118	2,3′,4,4′,5-pentachlorobiphenyl	29.6 (1.31)
C136	2,2′,3,3′,6,6′-hexachlorobiphenyl	17.1 (0.53)
C138	2,2′,3,4,4′,5′-hexachlorobiphenyl	25.1 (0.61)
C143	2,2′,3,4,5,6′-hexachlorobiphenyl	24.7 (0.88)
C153	2,2′,4,4′,5,5′-hexachlorobiphenyl	56.7 (3.07)
C155	2,2′,4,4′,6,6′-hexachlorobiphenyl	50.0 (1.0)
C167	2,3′,4,4′,5,5′-hexachlorobiphenyl	19.2 (2.75)
C180	2,2′,3,4,4′,5,5′-heptachlorobiphenyl	7.9 (1.64)
C185	2,2′,3,4,5,5′,6-heptachlorobiphenyl	<1
A1242[b]		887 (67)
A1254[c]		662 (62)
total PCB		2167 (34)

[a] IUPAC, International Union of Pure and Applied Chemists. [b] Aroclor 1242. [c] Aroclor 1254.

The constancy of K_d is of practical significance to preproject evaluation of disposal alternatives for PCB-contaminated dredged material. In particular, the long-term constancy of K_d during leaching of contaminated dredged material in confined disposal facilities (CDFs) has not been investigated. The purpose of this investigation was to study the factors affecting PCB distribution coefficients in sediment from the New Bedford Harbor Superfund Site, New Bedford, MA. Sediments were extracted sequentially in batch tests with distilled–deionized and saline waters, while the effects of conductivity, dissolved organic carbon, and numbers of microorganisms on PCB distribution coefficients were evaluated.

Materials and Methods

Extraction tests were conducted on a subsample of a very large (approximately 4000 L) composite sediment sample collected from the upper Acushnet River Estuary, New Bedford, MA, as described by Averett (*17*). The composite sediment sample had a specific gravity of 2.35, total organic carbon concentration of 2.6%, and a particle size distribution of 39% sand (>50 μm), 61% silt (2–50 μm), and 0% clay (<2 μm). Concentrations of selected PCB congeners, Aroclors, and total PCBs in New Bedford Harbor sediment are presented in Table I. Congener 7 was lowest in concentration (0.56 mg/kg) and congener 52

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was highest (177 mg/kg). Total PCB concentration was 2167 mg/kg. Batch tests showed no significant difference in extracted PCB concentrations for shaking times of 24, 48, and 168 h (*18*). All testing was conducted under anaerobic conditions. Anaerobic conditions were maintained because the leaching environment in a confined disposal facility is predominantly anaerobic for many years due to the high oxygen demand of contaminated sediments.

Sequential Batch Extraction. Wet sediment, mechanically mixed under a nitrogen atmosphere, was added to 450-mL stainless centrifuge tubes that had been double rinsed with acetone. Sufficient distilled–deionized water was added to give a sediment solids concentration of 250 g/L. Testing had shown that 250 g/L was the highest solids concentration that could be used and obtain the quantities of extractant needed for chemical analysis (*18*). Tubes were sealed, placed in a rotary tumbler (*19*), and turned end over end at 40 revolutions per minute for 24 h. Once removed from the tumbler, tubes were centrifuged for 30 min at 6000*g* and supernatants filtered through Whatman GF/D prefilters and Gelman AE glass fiber filters (nominal pore size of 1.0 μm). All filters were combusted at 400°C prior to use. Filtration was conducted under a nitrogen atmosphere. Filtrates were acidified with 1 mL of concentrated HCL to prevent precipitation and scavenging of PCB from solution by ferric iron precipitates. Filtrates were stored in the dark in acetone-rinsed 2-L glass bottles until analyzed for PCBs. Separate subsamples were acidified with sulfuric acid and set aside for analysis of dissolved organic carbon (DOC).

Following centrifugation and filtration, water was added by weight to the sediment remaining in the centrifuge tubes to replace that removed, and the shaking–solids separation procedures were repeated until sevel sequential extraction cycles had been completed. Two series of sequential extraction tests were conducted, one using distilled–deionized water and another using 20 parts per thousand saline water. The saline water was prepared in the laboratory by the method of Burkholder (*20*). Each series of sequential batch extraction tests was conducted in triplicate.

Sodium Metaphosphate Extraction. Wet sediment was also extracted in triplicate by using a 5% sodium metaphosphate solution in place of distilled or saline water. Procedures were identical with those described for sequential batch extraction except that only one extraction cycle was conducted.

Enumeration of Microorganisms. The numbers of microorganisms in the extractant were determined by use of a procedure based on the conventional plate count method (*21*). Following filtration at the end of each extraction cycle, a 2.1-mL aliquot from each of the three replicate extraction samples was carried through a dilution series from 10^0 to 10^9 with appropriate aseptic technique. The dilute was a sterile phosphate buffer (Butterfield's) containing 68 g of KH_2PO_4/L of glass-distilled water; the pH of the buffer was adjusted to 7.2 with 1 N NaOH. Each dilution was mixed with sterile melted Standard Methods Agar at 45 °C by the pour plate technique. Colonies were counted after incubations of 3–5 days at room temperature.

Chemical Analysis. Extractant and sediment samples were analyzed for PCBs as Aroclors, total PCBs (quantitated by using a multi-Aroclor standard), and selected PCB congeners by use of a Hewlett-Packard 5880A gas chromatograph equipped with an electron capture detector. Concentrations of PCB compounds in sediment were determined following Soxhlet extraction (50% hexane–50% acetone by volume) for 16 h and silica gel cleanup. Concentrations of PCB compounds in extractant were determined following methylene chloride extraction and silica gel cleanup. Average analytical reproducibility for PCB concentrations in extractions from replicate experiments was ±13%. Dissolved organic carbon in extractant samples was determined with an Oceanographic International 524B organic carbon analyzer.

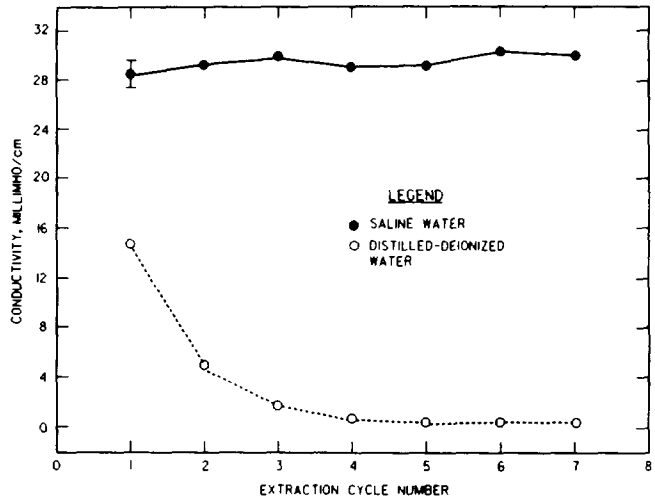

Figure 1. Conductivity during sequential leaching with distilled–deionized and saline water.

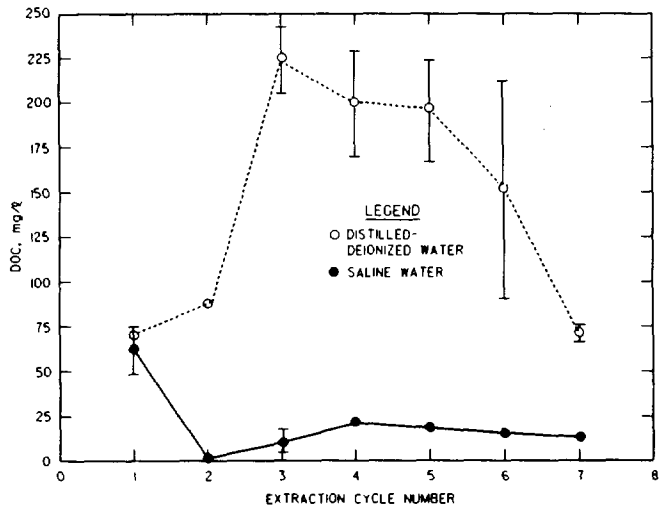

Figure 2. DOC during sequential leaching with distilled–deionized and saline water.

Results

Conductivity, DOC, and microbial colony-forming untis released during distilled–deionized and saline water extraction of anaerobic New Bedford Harbor sediment are shown in Figures 1–3, respectively. The conductivity curve for distilled–deionized water (Figure 1) indicates reduction of salinity and ionic strength as sequential extraction proceeded. Conductivity, as expected, remained constant during saline water sequential extraction. DOC concentrations (Figure 2) during distilled–deionized water extraction peaked during the third extraction cycle. DOC concentrations during saline water extraction did not change substantially during the course of sequential extraction. The elution of microbial colony-forming units (Figure 3), a component of DOC, during distilled–deionized water extraction was similar to DOC elution; however, the peak concentration occurred during the fifth extraction cycle.

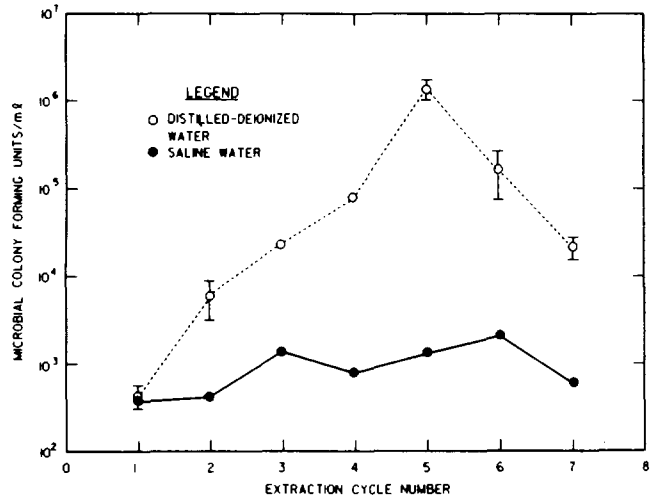

Figure 3. Numbers of microbial colony-forming units during sequential leaching with distilled–deionized and saline water.

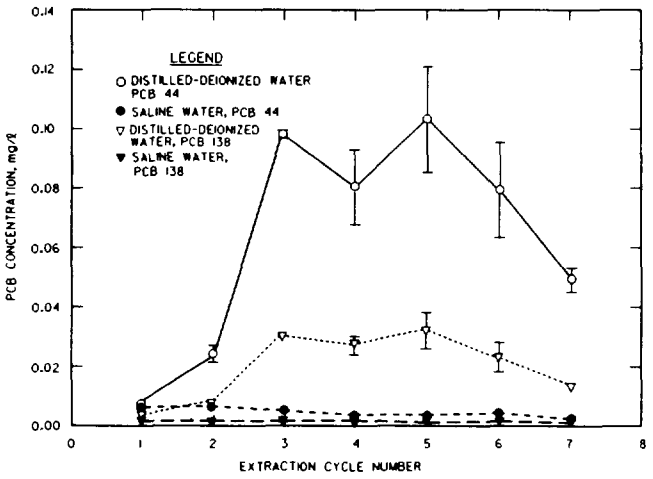

Figure 4. PCB congeners 44 and 138 during sequential leaching with distilled–deionized and saline water.

PCB results for distilled–deionized water and saline water extract PCB concentration followed the same trends as extraction of DOC and microorganisms. Aqueous-phase PCB concentrations exceeded pure water solubility limits during distilled water extraction for the great majority of extraction cycles. Aqueous-phase concentrations of PCB congeners C44 and C138 in extracts are presented in Figure 4. Aroclors, total PCBs, and most of the other PCB congeners showed the same general trend during sequential batch extraction as shown in Figure 4. The exceptions were for PCB congeners C50, C105, and C183. These PCB congeners did not extract at concentrations above the detection limit (0.001 mg/L). As shown in Figure 4, PCB concentrations during distilled–deionized water extraction increased during the first three cycles and then remained relatively constant during the third and fifth extraction cycles. PCB concentrations then declined during the sixth and seventh extraction cycles. PCB concentrations during saline water extraction did not change substantially during the course of sequential extraction.

The data in Figure 4 indicate nonconstant partitioning during sequential extraction with distilled–deionized water. Had partitioning been constant, PCB concentrations would have remained relatively constant or tended to decline, K_ds for selected PCB congeners (C8, C44, and C138) versus extractions cycle are shown in Figure 5. K_ds for other PCB congeners, Aroclors, and total PCB showed the same general trend as shown in Figure 5 for PCB congeners C8,

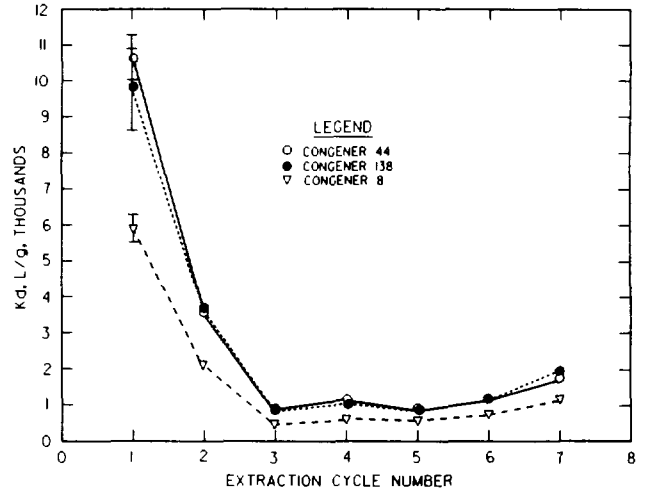

Figure 5. K_ds for PCB congeners 8, 44, and 138 during sequential leaching with distilled–deionized water.

C44, and C138. K_ds declined with successive extraction of the sediment solids up to extraction cycle 3. The decrease in K_d from extraction cycles 1–3 corresponds to decreasing conductivity and increasing concentrations of DOC and microorganisms in the extracts. From extraction cycles 3–5, K_d was relatively constant, corresponding to the development of peak concentrations of DOC and microorganism. The increase in K_d after extraction cycle 5 corresponds to decreases in extractant DOC concentrations and microbial colony-forming units. In contrast, K_ds for saline water extractions were relatively constant over the entire sequential extraction cycle.

The distribution coefficients determined in this study are operationally defined by the solids–liquid separation technique described in Materials and Methods. In this technique, C_w (eq 1) includes PCB adsorbed to DOC. Thus, K_d is not defined in terms of free or truly dissolved PCB concentration. Although DOC-bound PCB is not truly dissolved, this material can be advected by porewater flow and, therefore, transported as if dissolved. Further, leachate monitoring programs typically use a solids–liquid separation technique similar to the one employed here to determine "dissolved" contaminant concentrations. Since, K_d is an important parameter for preproject assessment of potential leaching problems, our method is justified in terms of relevant field transport mechanisms and the practical aspects of field monitoring.

Discussion

Ionic Strength Effects. Nonconstant PCB partitioning during sequential extraction of New Bedford Harbor sediment using distilled–deionized water is related to extraction of sediment salts. Salinity dependency can be qualitatively explained by the Gouy–Chapman model of charge distribution in double layers. According to the Gouy–Chapman model, decreasing ionic strength increases repulsive forces between layers (*22*) so that flocculated colloidal matter becomes dispersed. As salt is removed from estuarine sediment, the ionic strength is reduced and deflocculation of sediment organic carbon occurs, increasing the concentration of dissolved organic carbon. PCB bound to the colloidal matter is thereby mobilized. As a result, K_d decreases until the ionic strength is stabilized. Other investigators have also suggested that colloidal organic matter can act as a carrier of hydrophobic organics (*23–28*).

Release of DOC and microorganisms associated with reduction in sediment salinity during distilled–deionized

Table II. Concentrations [mg/L (Standard Error)] of PCBs in Distilled Water, Saline Water, and 5% Sodium Metaphosphate Leachate following 24 Hours of Shaking

	treatment		
compd	distilled water	saline water	5% sodium metaphosphate
C07	<0.001	<0.001	0.00012 (0.0003)
C08	0.029 (0.002)	0.020 (0.002)	0.095 (0.016)
C28	0.021 (0.0005)	0.025 (0.002)	0.11 (0.03)
C44	0.008 (0.0006)	0.004 (0.0006)	0.03 (0.007)
C49	0.003 (0.0007)	0.002 (0.00)	0.10 (0.02)
C52	0.024 (0.002)	0.015 (0.001)	0.013 (0.003)
C70	0.005 (0.00)	0.004 (0.0003)	0.02 (0.009)
C77	0.009 (0.009)	0.010 (0.002)	0.0034 (0.0008)
C82	0.0003 (0.0003)	0.002 (0.00)	0.02 (0.005)
C87	<0.001	<0.001	0.0013 (0.0001)
C97	0.003 (0.00)	0.002 (0.00)	0.013 (0.003)
C101	0.007 (0.002)	0.006 (0.0003)	0.033 (0.007)
C118	0.004 (0.0003)	0.003 (0.00)	0.135 (0.003)
C136	0.001 (0.00)	0.002 (0.0003)	0.010 (0.007)
C138	0.003 (0.0003)	0.001 (0.00)	0.006 (0.001)
C143	<0.001	<0.001	0.011 (0.003)
C153	0.005 (0.001)	0.004 (0.00)	0.024 (0.007)
C155	<0.001	0.004 (0.0003)	0.02 (0.004)
C167	<0.001	<0.001	0.0015 (0.0003)
C180	<0.001	<0.001	0.0016 (0.0003)
Aroclor 1242	0.203 (0.007)	0.183 (0.012)	0.80 (0.14)
Aroclor 1254	0.137 (0.02)	0.083 (0.003)	0.39 (0.13)
total PCB	0.327 (0.003)	0.287 (0.024)	1.18 (0.22)

water extraction is probably the reverse of the salt flocculation process that occurs in estuaries. Transport of colloidal material from freshwater into estuaries is accompanied by salinity-dependent flocculation and deposition (*29–32*). Flocculation is a reversible process when sediment salinity decreases (*33*).

PCB mobilization, facilitated by release of organic carbon and microparticulates associated with sediment solids, is indicated by extraction results (one extraction cycle) obtained with a dispersant (5% solution of sodium metaphosphate). Results of the sodium metaphosphate extractions are summarized in Table II along with equivalent first extraction cycle PCB concentrations for distilled and saline water extracts. As shown in Table II, saline and distilled water PCB extract concentrations were approximately the same in the first extraction cycle when conductivity in the distilled–deionized water extract was sufficient to suppress release of flocculated organic carbon and microparticulates. However, much higher PCB concentrations were observed in the distilled–deionized water extract containing the dispersant. In addition, when conductivity of the extractant remained constant, i.e., in the saline water sequential batch extraction tests, no mobilization of PCB or DOC and no change in level of microorganisms occurred.

Modeling Implications. A three-phase partitioning model in which PCB is partitioned between sediment organic carbon, DOC, and water is a better approach to modeling equilibrium partitioning than the simple model given in eq 1 (*16, 27, 28*). Gschwend and Wu (*16*) used a three-phase partitioning model to show that PCBs sorbed to nonfilterable particulate matter in the "dissolved" phase can account for the dependency of K_d on solids concentrations. Nonconstancy is introduced when the liquid phase contains DOC and microparticulates that cannot be removed and whose concentration and perhaps composition changes with solids concentration. As previously discussed, our data suggest that DOC-bound PCB explains the differences between our distilled–deionized and saline water sequential batch extraction data.

DOC-bound PCB also explains the nonconstant partitioning during sequential extraction with distilled–deionized water. Rearrangement of eq 9 from Gschwend and Wu (*16*) yields the following:

$$(K_{oc\text{-}p}{}^{obsd})^{-1} = (K_{oc\text{-}p}{}^{true})^{-1} + \frac{K_{oc\text{-}NSP}{}^{true}}{K_{oc\text{-}p}{}^{true}}[DOC] \quad (2)$$

where $K_{oc\text{-}p}{}^{true}$ is the partition coefficient for truly dissolved PCB and sediment organic carbon (L/kg), $K_{oc\text{-}p}{}^{obsd}$ is the observed partition coefficient for dissolved PCB and sediment organic carbon (L/kg), and $K_{oc\text{-}NSP}{}^{true}$ is the partition coefficient for truly dissolved PCB and DOC (L/kg). In eq 2, we have assumed that DOC is a satisfactory measure of sorbing materials that pass a 1-μm filter. Gschwend and Wu (*16*) refer to these materials as nonsettling particulates (NSPs).

Table III. Coefficients for Functional Dependence of $(K_{oc\text{-}p}{}^{obsd})^{-1}$ on DOC[a,b]

PCB	intercept, kg/L	slope	r^2
C8	(7.94×10^{-6})	0.167	0.68
C28	(2.0×10^{-6})	0.298	0.76
C44	(1.55×10^{-6})	0.164	0.68
C52	(8.13×10^{-7})	0.149	0.71
C70	1.45×10^{-6}	0.113	0.65
C77[c]	(4.90×10^{-7})	0.161	0.76
C97	2.95×10^{-6}	0.183	0.57
C101	2.04×10^{-6}	0.166	0.69
C138	(3.63×10^{-8})	0.127	0.73
C153	9.14×10^{-7}	0.151	0.64
total PCB[d]	4.10×10^{-6}	0.179	0.64

[a] $n = 21$. [b] Intercepts constrained to be $>1/K_{ow}$; values in parentheses indicate the constraint was the best fit. [c] K_{ow} for PCB C66 used as constraint on the intercept. [d] Intercept constrained to be >0.

Coefficients for the functional dependency of $(K_{oc\text{-}p}{}^{obsd})^{-1}$ on DOC concentration as modeled in eq 2 are presented in Table III for congeners with complete data sets (detectable quantities of PCBs in each cycle of the extraction procedures; $n = 21$) and total PCBs. The coefficients provided in Table III are specific for the sediment tested and the experimental methods used in this study and therefore are not likely to be applicable to other sediments. Data and best-fit lines are shown in Figure 6 for selected PCB congeners and total PCBs. The trends in Figure 6 suggest that $K_{oc\text{-}p}{}^{obsd}$ is controlled largely by DOC. The regression coefficients (r^2 values) in Table III show relatively good fit for the Gschwend and Wu model. Deviation of PCB data from eq 2 may be due to changes in the nature of DOC as well as changes in DOC concentration with continued extraction of DOC, resulting in modifications of sorbent surfaces. In addition, the dissolved organic matter measured in the extracts is a mixture of many natural organic compounds that may act selectively and differently with regard to deflocculation and sorption.

In the regressions used to develop the coefficients provided in Table III, the intercepts for the PCB congeners were constrained to be greater than or equal to $(1/K_{ow})^{-1}$, where K_{ow} is the octanol–water partition coefficient reported by Rapaport and Eisenreich (*34*). For total PCB, the intercept was constrained to be greater than zero. The octanol–water partition coefficient is the upper bound on $K_{oc\text{-}p}{}^{true}$ since studies (*5–7*) have shown that K_{oc} is usually less than K_{ow}. The intercepts in parentheses in Table III indicate the fitted intercept was exactly $1/K_{ow}$. Unconstrained regressions yielded slightly negative intercepts for many of the PCB congeners of magnitude 1×10^{-7} or less, with no improvement in the regression coefficients. Since

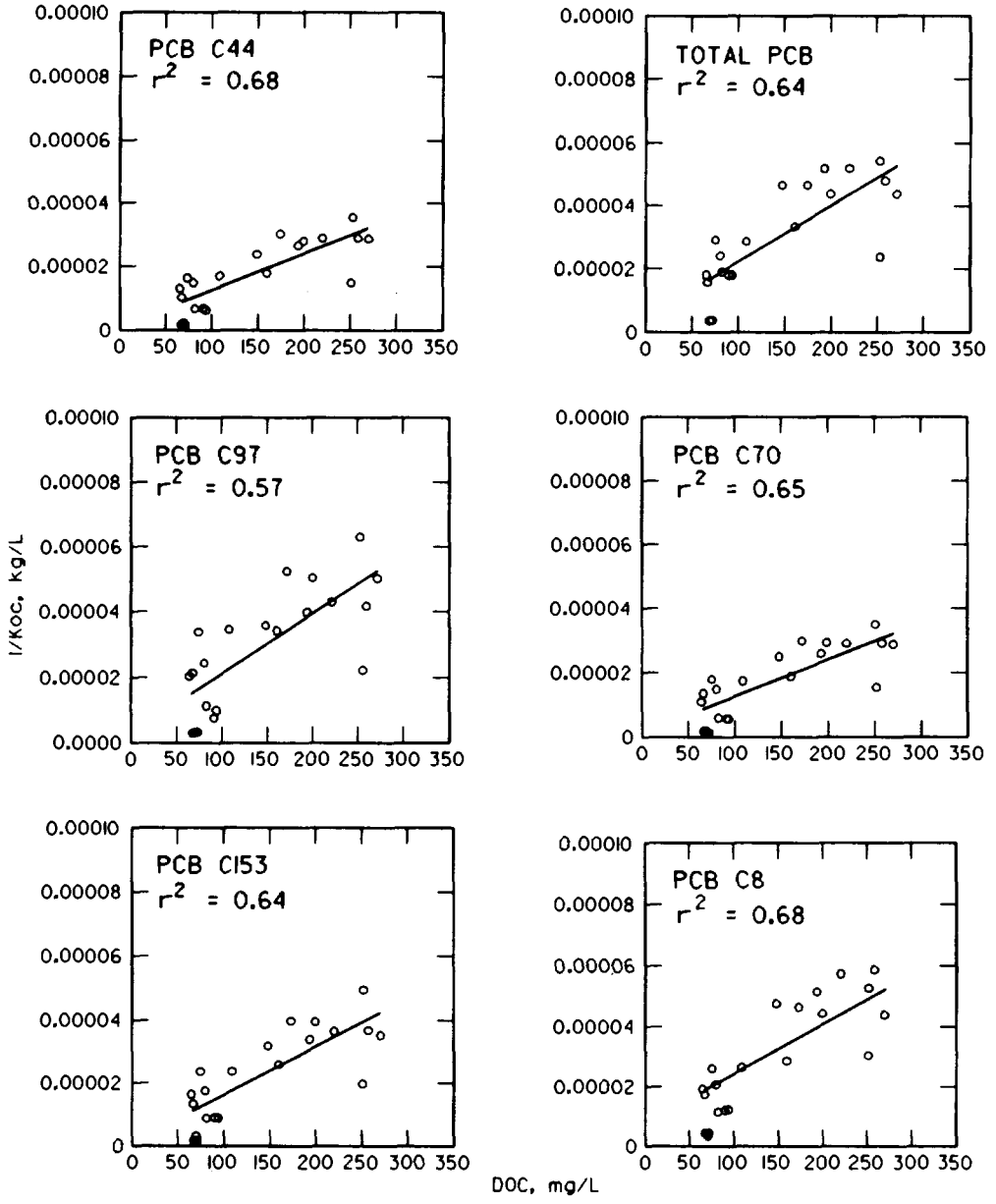

Figure 6. Data and lines of best fit for Gschwend and Wu model.

the scatter in the data make it difficult to estimate $K_{oc\text{-}p}^{true}$, the intercepts in Table III should not be interpreted as statistically meaningful estimates of $K_{oc\text{-}p}^{true}$.

Best-fit slopes were not sensitive to constraints placed on the intercepts; that is, the slopes provided in Table III are nearly identical with those obtained without constrained intercepts. Since the slopes are estimators of $K_{oc\text{-}NSP}^{true}/K_{oc\text{-}p}^{true}$, they represent the relative sorption potential of DOC and sediment organic carbon for PCBs. As shown in Table III, the slopes were generally between 0.1 and 0.3 with most of the values between 0.15 and 0.19. These data indicate that the materials that comprise DOC have lower sorption potential than the sediment organic carbon for PCBs. This suggests that the organic carbon that becomes dispersed in the aqueous phase is more polar than the organic carbon than remains in the sediment phase and is, as a result, less efficient at sorbing nonpolar PCBs (*35*, *36*).

The Gschwend and Wu model tended to overpredict $K_{oc\text{-}p}^{true}$, but showed the correct trend for DOC-dependent partitioning. In spite of the problems with fitting intercepts, which we believe are due to scatter in the data, the model shows that changing DOC concentration was the primary cause of nonconstant partitioning.

Conclusions

Sequential batch extraction tests showed that leaching of anaerobic New Bedford Harbor sediment with distilled–deionized water resulted in greater mobilization of PCBs than leaching with saline water. During this mobilization, conductivity- (salinity-) dependent PCB partitioning was evident. The strong relationships between conductivity, DOC concentration, and PCB concentration in extractants indicate that salinity-dependent release of sediment organic carbon was the mechanism controlling nonconstant PCB partitioning.

Although we did not specifically analyze colloidal PCB in New Bedford Harbor, sediment, our data are consistent with the hypothesis that organic colloids and microparticulates that comprise naturally occurring DOC significantly affect the environmental mobility of PCBs. The data in this study indicate that three-phase partitioning models may be needed to adequately describe PCB leaching from contaminated estuarine sediments and dredged material.

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Registry No. PCB 7, 33284-50-3; PCB 8, 34883-43-7; PCB 28, 7012-37-5; PCB 44, 41464-39-5; PCB 49, 41464-40-8; PCB 50, 62796-65-0; PCB 52, 35693-99-3; PCB 70, 32598-11-1; PCB 77, 32598-13-3; PCB 82, 52663-62-4; PCB 87, 38380-02-8; PCB 97, 41464-51-1; PCB 101, 37680-73-2; PCB 105, 32598-14-4; PCB 118, 31508-00-6; PCB 136, 38411-22-2; PCB 138, 35065-28-2; PCB 143, 68194-15-0; PCB 153, 35065-27-1; PCB 155, 33979-03-2; PCB 167, 52663-72-6; PCB 180, 35065-29-3; PCB 185, 52712-05-7; Aroclor 1242, 53469-21-9; Aroclor 1254, 11097-69-1; carbon, 7440-44-0.

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