

Partitioning of Polychlorinated Biphenyl Congeners in the Seawater of New Bedford Harbor, Massachusetts[†]

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The partitioning of polychlorinated biphenyls (PCBs) between particulate and dissolved phases of seawater was examined along a concentration gradient in New Bedford Harbor, MA. Regression analysis was used to examine the relationship between the log of the partition coefficients (K_p) and the log of the octanol/water partition coefficients (K_{ow}) for six PCB congeners at four stations. A seasonal trend (i.e., summer–winter) in partitioning was observed. Log K_p was inversely correlated with temperature and to a lesser extent with total suspended solids (TSS) at each station. Multiple regression analysis indicated that approximately 70% of the variability in log K_p along the PCB concentration gradient was accounted for by temperature, log K_{ow} , and TSS. Temperature and log K_{ow} were equally important in defining changes in log K_p , with TSS contributing the least predictive ability.

Introduction

Partitioning of organic contaminants between dissolved and particulate phases of the water column is a major process controlling their fate, transport, and bioavailability in aquatic systems. The fate of hydrophobic organic contaminants in natural systems is highly dependent on their sorptive behavior due to differences in transport mechanisms for water and particulates (1). The transport of large particles in an estuary is determined by local hydrodynamic characteristics and settling rates. Organic contaminants freely dissolved or bound to small nonsettling particles and organic matter may be transported from estuaries to the open ocean. In addition, it is generally accepted that freely dissolved hydrophobic pollutants are more bioavailable than those bound to particles.

In order to model the partitioning of contaminants, researchers have related the octanol/water partition coefficients (K_{ow} s) of various hydrophobic organic contaminants with their particulate/water partition coefficients (K_p s). This relationship is based on the premise that the manner in which an organic compound partitions between particulate organic matter and water should correlate well with its partitioning between water and octanol, an immiscible organic solvent (2).

However, many factors affect the partitioning of contaminants between the dissolved and particulate phases of seawater. Perhaps the most widely examined component affecting partitioning coefficients is the "solids concentration effect" (3–5). Studies have shown that K_p and total suspended solids (TSS) are inversely related (6). Several theories have been put forth to explain this

observation: (1) existence of a colloidal phase, associated with the dissolved, which increases when particles increase (7); (2) particle interactions inducing desorption of labile-sorbed contaminants due to increased particle collisions (8); and (3) increased aggregation at high particle concentration decreasing surface area and increasing the diffusive path length, thus affecting kinetics (9). In addition to TSS, partition coefficients have been shown to be influenced by particle organic carbon content (1, 6), particle size (1), salinity, and pH (10).

Definition of the effect of these variables on contaminant partitioning in natural systems is difficult because the majority of research on partition coefficients has been completed under laboratory conditions, which often employ large particle concentrations (10^2 – 10^5 mg/L) and artificial media. Therefore, results may not extrapolate well to natural systems where particle concentrations are much lower (10^0 – 10^2 mg/L). Also, the surface layer of in situ water-borne particles such as algae and detritus may contain much more organic matter than the sediments, soils, and sands used in laboratory experiments. For these reasons, many scientists have expressed a need for the increased field study of partitioning behavior. The major focus of this study was to determine how octanol/water partition coefficients (K_{ow}) relate to field-generated particulate/water partition coefficients (K_p), thereby providing an evaluation of the ability of the existing partitioning theory to predict the partitioning of PCBs under field conditions.

PCBs were chosen for this study because they are resistant to degradation, are hydrophobic, and have the analytical capability for detection at low levels. PCBs occur as a series of 209 chlorinated compounds with distinct physicochemical properties, including partition coefficients. Also, a major study being conducted at a highly PCB contaminated estuary, New Bedford Harbor (NBH), MA, afforded an opportunity to obtain and analyze numerous dissolved and particulate water samples.

Historically, NBH has received high levels of anthropogenic inputs from a variety of sources, including significant amounts of PCBs. Sediment PCB concentrations have been reported as high as 100 000 $\mu\text{g/g}$, and water column PCB concentrations within the harbor are in the low microgram per liter range (11). These factors have resulted in areas of the harbor being placed on the National Priority List as a Superfund site. As part of the NBH remediation process, a pilot dredging project was conducted to determine the efficacy of removing PCB-contaminated sediments from the harbor (12). A corresponding monitoring plan was designed and implemented to determine both chemical and biological effects of this project (13). Seawater was monitored for several param-

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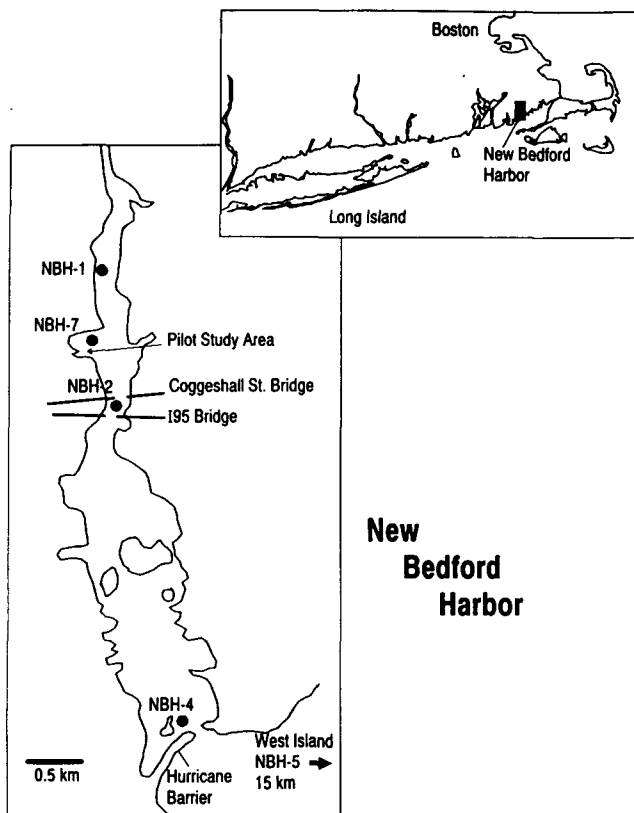


Figure 1. Map of New Bedford Harbor and Buzzards Bay showing the location of the four stations used in this study.

eters, including levels of particulate and dissolved PCBs, at three stations in the harbor and at a reference site in Buzzards Bay. Data were collected along a PCB concentration gradient (concentration decreased by 2 orders of magnitude), thus providing for an evaluation of partitioning with respect both to changing PCB concentration and other environmental variables.

The objectives of this paper include (1) determination of K_{ps} for selected PCB congeners along a contamination gradient in the field, (2) examination of the relationship between field generated K_{ps} and K_{ows} , and (3) effect of selected environmental factors (i.e., temperature, suspended solids) on K_p under field conditions.

Materials and Methods

Sample Collection. A detailed description of the monitoring program, including sample collection and station selection strategies, was presented by Nelson and Hansen (13). Briefly, at stations NBH-1, NBH-2, and NBH-4 (Figure 1), 1 L of seawater was pumped manually from each of three depths (1 m below the surface, middepth, 1 m above the bottom) and composited into one sample. This sampling occurred at five equally spaced time intervals over each tidal cycle. Immediately after each of the five collections, a 200-mL subsample was filtered through a 47-mm Gelman Type A/E glass fiber filter ($\sim 1\text{-}\mu\text{m}$ particle size retention). The five filtrates were composited and stored in a combusted (muffled for 6 h at 450°C) glass bottle at 4°C . The filter was placed in combusted aluminum foil and stored at 4°C .

Because of lower PCB concentrations at station NBH-5, a high-volume seawater sampling device was utilized (14). Thirty-six liters of seawater was pumped from a

depth of 3 m (middepth) then passed through a 293-mm Gelman Type A/E glass fiber filter and a cartridge containing polyurethane foam plugs.

Sample Extraction. Detailed sample extraction procedures were presented by Bergen (15). Exact volumes were recorded for the dissolved phase water samples, octochloronaphthalene (OCN) was added as an internal standard, and extraction was carried out with freon (1,1,2-trichlorotrifluoroethane). Extracts were volume-reduced and exchanged to heptane, partitioned against sulfuric acid to remove organic interferences, and stored at 4°C .

Foam plug samples were extracted with acetone and hexane. OCN was used as an internal standard and was added to the organic extracts after the extraction of the plugs. The extracts were volume-reduced, solvent-exchanged to heptane, partitioned against concentrated sulfuric acid overnight, and stored at 4°C .

For filter samples, OCN was added as an internal standard followed by extraction with acetone and freon using a Polytron homogenizer. Samples were centrifuged, and the supernatant was decanted into a 1-L separatory funnel and extracted with freon. The extract was volume-reduced, solvent-exchanged to 1 mL of heptane, and treated with concentrated sulfuric acid. The heptane layer was drawn off and stored at 4°C .

Sample Analysis. Heptane extracts were analyzed for PCBs by electron capture-capillary gas chromatography (EC-GC). One microliter of each sample was injected into a Hewlett-Packard 5890 gas chromatograph equipped with a splitless injector, 30-m DB-5 fused silica capillary column (0.25- μm film thickness, 0.25-mm i.d., J&W Scientific, Folsom, CA) and an electron capture detector. Injection port temperature was set at 270°C . Column temperature was held at 60°C for 1 min, then heated to 315°C at a rate of $10^\circ\text{C min}^{-1}$, and held for 13.5 min. The detector temperature was 325°C . Analog data from the GC were digitized using a Perkin-Elmer 3200 computer equipped with Perkin-Elmer LIMS/CLAS software. Individual congeners were quantified against the OCN internal standard. PCB standards were obtained as powders in pure form from Ultra Scientific, North Kingston, RI.

Blank analyses were conducted for each sample type and for every other set of six samples analyzed. In all cases, results for blanks showed none or only trace levels of PCB congeners. Spike and recovery data indicated that analyte recovery ranged from 85 to 100% with these methods.

Data Analysis. From July 1987 to October 1990, dissolved and particulate water samples were collected at stations NBH-1, NBH-2, NBH-4, and NBH-5. Thirteen individual congeners were quantitated in each sample; however, only six congeners were chosen for consideration in this report (Table I). Selection was based on the fact that only these six congeners were present in detectable amounts in all the 1-L dissolved-phase seawater samples. The congeners studied included a range of chlorination levels (two each of tetra-, penta-, and hexachlorobiphenyls) and log K_{ow} values (5.84–6.92).

Because of differing terminology in the literature, the variables used to calculate partition coefficients in this paper were operationally defined as particulate (nonfilterable) and dissolved (filterable through a Gelman Type A/E glass fiber filter, retention size approximately $1.0\text{-}\mu\text{m}$). Additional variables measured during the study on a daily basis included temperature and TSS. Salinity was not

Table I. Mean (Standard Deviation) Partition Coefficients (K_p) for Six PCB Congeners at Four Stations in New Bedford Harbor, MA^a

| congener | PCB congener identification ^b | low K_{ow} ^c | NBH-1, $n = 17$ | NBH-2, $n = 33$ | NBH-4, $n = 28$ | NBH-5, $n = 9$ |
|----------|--|---------------------------|-----------------|-----------------|-----------------|----------------|
| CB052 | 2,2',5,5'-TCB | 5.84 | 5.15 (0.36) | 5.07 (0.31) | 5.01 (0.43) | 4.73 (0.25) |
| CB047 | 2,2',4,4'-TCB | 5.85 | 5.64 (0.42) | 5.53 (0.35) | 5.47 (0.38) | 5.18 (0.23) |
| CB101 | 2,2',4,5,5'-PeCB | 6.38 | 5.35 (0.41) | 5.17 (0.32) | 5.04 (0.35) | 4.87 (0.29) |
| CB118 | 2,3',4,4',5-PeCB | 6.74 | 5.82 (0.49) | 5.73 (0.39) | 5.71 (0.36) | 5.55 (0.26) |
| CB138 | 2,2',3,4,4',5'-HxCB | 6.83 | 5.89 (0.44) | 5.82 (0.41) | 5.72 (0.36) | 5.52 (0.50) |
| CB153 | 2,2',4,4',5,5'-HxCB | 6.92 | 5.91 (0.48) | 5.81 (0.43) | 5.84 (0.33) | 5.65 (0.29) |

^a Values for stations NBH-1, NBH-2, and NBH-4 include summer and winter sampling events (n). Values at NBH-5 include only summer samples. ^b Ballschmiter and Zell (16). ^c Hawker and Connell (17).

measured daily; therefore, it is not discussed as a modifying variable. However, previous measurements have shown little variation in salinity among stations.

Statistical analyses were completed using programs from the Statistical Analysis System (SAS) Institute Inc. (18). Correlation coefficients between variables were computed using Pearson product-moment and weighted product-moment programs. Linear and stepwise regression analyses, analysis of variance (ANOVA), and analysis of covariance also were employed in the synthesis of these data.

Results and Discussion

Partitioning Coefficients. Mean K_p s were calculated for six selected PCB congeners at each station over the entire study period (Table I). At all stations, K_p s generally increased with congener K_{ow} s with the notable exception of CB047. The data showed an apparent trend of higher K_p along the PCB contamination gradient moving from NBH-1 (highest PCBs) to NBH-5 (lowest PCBs); however, ANOVA analysis revealed that only station NBH-5 K_p s were statistically different.

Relationship between K_p and K_{ow} . The major focus of this study was to examine the relationship between field-generated K_p s and K_{ow} s for several PCB congeners. The observed congener K_p s correlated with K_{ow} s at the four stations with varying degrees of strength. The regression coefficient, r^2 , increased along the PCB concentration gradient from the most contaminated station, NBH-1 ($r^2 = 0.29$), to the least contaminated, NBH-5 ($r^2 = 0.58$). Analysis of covariance indicated that the slopes and intercepts relating $\log K_p$ to $\log K_{ow}$ were equal at stations NBH-1, NBH-2, and NBH-4 and different at NBH-5. Data were combined from the three equivalent stations into one equation relating $\log K_p$ and $\log K_{ow}$:

$$\log K_p = 0.688 \log K_{ow} + 1.074; \quad r^2 = 0.38, \quad N = 530 \quad (1)$$

This analysis indicated that K_{ow} alone only accounted for 38% of the observed variability in K_p . Results from other field validations of this approach have been mixed. Duinker (19) found an apparent linear relationship between $\log K_p$ and $\log K_{ow}$ for several PCB congeners in the North Sea. Eadie et al. (20) reported a good correlation ($r^2 = 0.84-0.94$) between K_{oc} (K_p normalized to organic carbon) and K_{ow} stating that "ambient Great Lakes POC is similar to octanol as a substrate for binding". However, Baker et al. (21) noted a "lack of dependence" of $\log K_p$ on $\log K_{ow}$ in the Great Lakes over an 8-year period. The present findings suggested that K_{ow} alone could not be used to model partitioning in New Bedford Harbor; therefore, other factors were investigated.

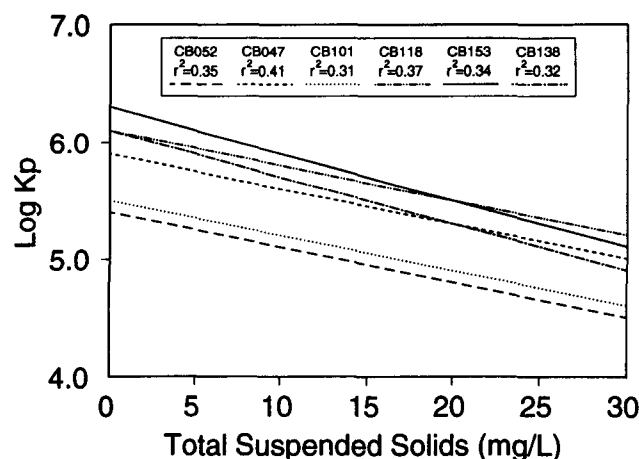


Figure 2. Relationship between $\log K_p$ and TSS (total suspended solids) for each of the six congeners evaluated in this study. Also shown are the regression coefficients for each line.

Factors Affecting K_p . An inverse relationship between $\log K_p$ and TSS occurred at each station; however, these correlations were not as strong as many laboratory studies have shown. Regression coefficients indicated that TSS alone could explain approximately 30–40% of the variability in K_p , depending upon the station and the congener (Figure 2). Similarly, Baker et al. (21) reported that TSS could explain 20–73% of the variability of $\log K_p$ in Lake Superior. Also, Duinker (19) found a weak inverse relationship between $\log K_p$ and TSS for various PCB congeners in the North Sea.

The other prominent environmental parameter that changed during this study was temperature. A significant inverse correlation was observed between $\log K_p$ and temperature. Analysis of covariance indicated that this relationship was the same at all stations; however, it differed between congeners (Figure 3). The relationship with temperature suggested that a seasonal trend in partitioning existed.

Therefore, data were sorted into summer (temperature range 17–26 °C) and winter (temperature range 1–11 °C) collections. Analysis of the summer data (covariance) indicated that all four stations exhibited similar relationships between $\log K_p$ and $\log K_{ow}$. These data were combined and one relationship established for the summer samples:

$$\log K_p = 0.640 \log K_{ow} + 1.188; \quad r^2 = 0.52, \quad N = 334 \quad (2)$$

Similar analysis of the winter data indicated that the relationship between K_p and K_{ow} was the same at the three stations sampled (no winter data at NBH-5). Combination of these data produced the following relationship (Figure

Table II. Seasonal Variation of Temperature and Total Suspended Solids (TSS) at Three New Bedford Harbor Stations*

| station | temp (°C) | | TSS (mg/L) | | particulate (C _p) PCB (μg/g) | | | |
|---------|------------|----------|------------|--------|--|-----------|-----------|-----------|
| | summer | winter | summer | winter | CB052 | | CB153 | |
| | | | | | summer | winter | summer | winter |
| NBH-1 | 23 [18-26] | 4 [1-8] | 14 (7) | 5 (2) | 3.1 (1.5) | 8.7 (4.8) | 1.3 (0.7) | 3.9 (1.9) |
| NBH-2 | 22 [18-25] | 4 [1-10] | 13 (8) | 5 (3) | 1.1 (0.4) | 2.3 (0.9) | 0.7 (0.3) | 2.0 (0.9) |
| NBH-4 | 21 [18-24] | 5 [1-11] | 15 (10) | 4 (2) | 0.2 (0.1) | 0.5 (0.1) | 0.2 (0.1) | 0.6 (0.2) |

* Temperature data are presented as mean and [range]. All other data show mean and (standard deviation). Also presented are the particulate PCB concentrations (μg/g) for two congeners.

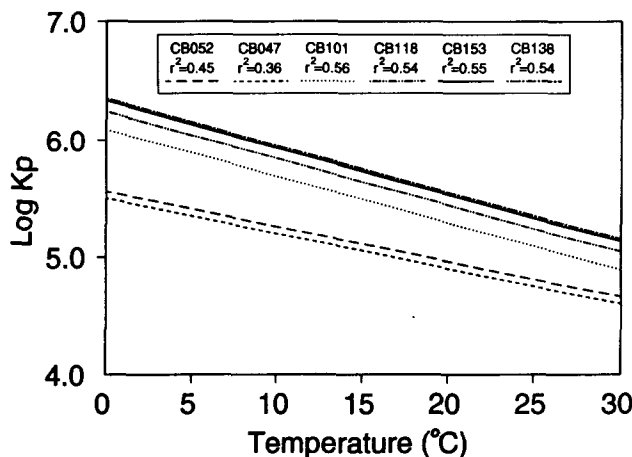


Figure 3. Relationship between log K_p and temperature for each of the six PCB congeners evaluated in this study. Also shown are the regression coefficients for each line.

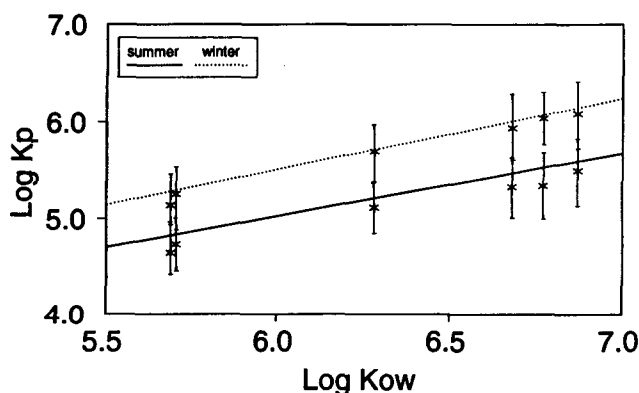


Figure 4. Relationship between log K_p and log K_{ow} , separated into summer and winter collections, for the six PCB congeners evaluated in this study. Bars show 1 SD about the mean for each congener. Summer data were collected at stations NBH-1, NBH-2, NBH-4, and NBH-5, with temperature ranging from 17 to 26 °C. Winter data were collected at stations NBH-1, NBH-2, and NBH-4, with temperature ranging from 1 to 11 °C.

4):

$$\log K_p = 0.733 \log K_{ow} + 1.125; r^2 = 0.52, N = 187 \quad (3)$$

Separation of the data by season resulted in a marked improvement in the regression coefficient from 0.38 to 0.52. No significant difference was found between the slopes of the lines for the winter and summer data; however, the intercepts were significantly different. This indicated that some seasonal factor(s) affecting the absolute coefficients, however, did not change the relative relationship between log K_p and log K_{ow} .

The individual variables used to calculate K_p , (dissolved (C_w) and particulate (C_p) PCB concentrations) were

examined with respect to TSS and temperature using Pearson correlation coefficients. C_w showed no correlation with either temperature or TSS. In contrast, C_p was correlated inversely with both TSS and temperature. Statistically significant differences were observed for all six mean C_p values between winter and summer samples. An example of this difference is presented for a lower (CB052) and higher (CB153) molecular weight congener in Table II. Although mean absolute TSS values (mg/L) were higher in the summer, C_p values, calculated on a per gram basis, were greater during the winter. Therefore, the elevation of K_p s in the winter and the particulate PCB concentrations appear to be interrelated.

In order to simultaneously evaluate the relative importance of each of these parameters on K_p , stepwise multiple regression analysis was conducted. Data from the three harbor stations yielded the following equation:

$$\log K_p = -0.01 \text{ TSS} + 0.69 \log K_{ow} - 0.03 \text{ temperature} + 1.6; r^2 = 0.70, N = 262 \quad (4)$$

| | TSS | log K_{ow} | temperature |
|-------|--------|--------------|-------------|
| r^2 | (0.04) | (0.34) | (0.32) |

The use of these three variables accounted for approximately 70% of the variability associated with congener K_p s along the PCB concentration gradient. The importance of temperature in predicting K_p was expected, given the seasonal dependence of the K_p/K_{ow} relationship discussed earlier. However, the partial regression coefficients indicated that TSS did not improve the fit of the data as much as temperature and K_{ow} , which were of equal importance.

As previously discussed, the dependence of log K_p on TSS was not as strong in this field study as many laboratory studies have suggested (3-5). This may be because TSS values in the present study were much lower than those used in most laboratory studies and also that TSS values were relatively similar along the PCB concentration gradient in this study. In addition, several authors have hypothesized the presence of a third phase in seawater, colloidal or nonsettling particles (NSPs), and state that this phase must be accounted for when evaluating partitioning and TSS effects (7, 19, 20, 22, 23). This colloidal phase was found to be increasingly important in areas of high dissolved organic carbon (DOC) and TSS (23). During the pilot study, DOC was not measured. However, subsequent water sampling in NBH has included some DOC samples. Preliminary results indicated that DOC was highest at the most contaminated station (NBH-1) and decreased significantly down the harbor (15). It is possible that differences in K_p observed between stations may relate to this third phase in seawater.

Use of the K_{ow}/K_p Relationship To Predict Field Concentrations. Additional dissolved and particulate

water samples are being collected currently in New Bedford Harbor to test the utility of eqs 2 and 3 for estimating the partitioning of PCBs in the field. However, as a preliminary exercise, the predictive ability of eqs 2 and 3 was tested by comparing estimated values with measured values from an independent whole-water database collected at station NBH-4. First, eqs 2 and 3 were used to calculate a summer and winter dissolved PCB concentration (as the sum of the six congeners) from the measured particulate concentrations. This step was repeated to estimate the particulate PCB concentration from the measured dissolved PCB concentration. The dissolved and particulate estimates were summed to obtain a total PCB concentration for the summer and winter and then compared to the measured PCB value from the independent whole-water samples.

The estimated summer and winter total PCB concentrations (0.0288 and 0.0251 $\mu\text{g/L}$, respectively) were within 22% and 15% of the measured summer and winter total PCB concentrations (0.0236 and 0.0295 $\mu\text{g/L}$, respectively) from the independent whole-water data set. The analytical uncertainty associated with direct water measurement of PCB congeners is approximately 15–25%; therefore, the estimated values were within this uncertainty. While additional data are required to test the validity of eqs 2 and 3 more rigorously, the relative agreement between the estimated values and the measured whole-water PCB concentrations is promising. These data indicate that, at least on a site-specific basis, equations can be developed to estimate PCB partitioning in the field.

Conclusions

The K_p s of six selected PCB congeners, measured along a concentration gradient that changed by 2 orders of magnitude, generally increased with the K_{ow} of the congener. Observed congener K_p s correlated with K_{ow} s with varying degrees of strength (strongest at the least contaminated station). Log K_p varied inversely with TSS; however, the correlation was not as strong as some laboratory studies have demonstrated. Depending on the congener, TSS alone accounted for 30–40% of the variability observed in K_p . Preliminary evidence suggested that this correlation improved at low dissolved organic carbon concentrations.

When considered with a number of parameters, log K_p in New Bedford Harbor was modeled best using temperature and log K_{ow} . Approximately 70% of the variability in log K_p along the PCB concentration gradient could be accounted for by log K_{ow} , temperature, and TSS, with TSS adding the least predictive ability. When subset by temperature (i.e., summer and winter), the relationship (slope) between log K_p and log K_{ow} was the same at all stations; however, the intercepts between summer and winter were different. Elevated K_p s were observed during the winter. Based on this study, temperature is an important variable that must be considered when modeling environmental contaminants in aqueous systems.

Estimated total PCB concentrations, based on the relationships generated in this paper, were within approximately 25% of direct total PCB concentrations quantified in an independent whole-water data set. The use of predictive equations to model field K_p s shows promise; however, further field investigations must be conducted to better gauge the range of conditions under which the relationships are applicable.

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