

# *In-situ* measurements of polychlorinated biphenyls in the waters of San Diego Bay

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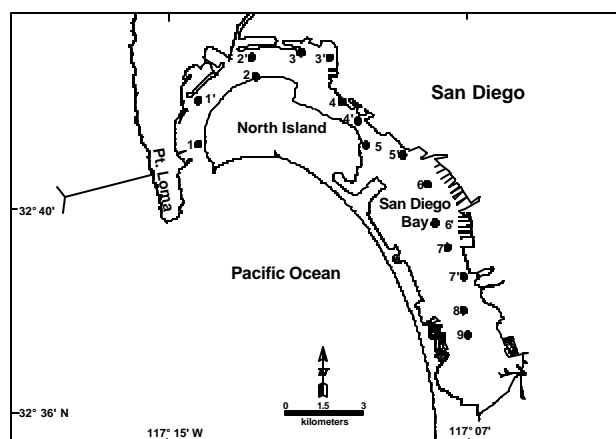
**ABSTRACT** - Sediments of San Diego Bay (SDB) are known to contain elevated levels of polychlorinated biphenyls (PCBs), but no studies have been directed toward assessing water column contamination. Measurements of PCB concentrations in the water column of SDB were taken with an *in situ* sampling system. Except for one sample collected near the bay mouth, all samples contained higher PCB concentrations than the 30-d average discharge limit established by the California Ocean Plan. The highest concentrations of water column PCBs occurred in the central bay area (Central Bay), consistent with the previous findings that sediments in the Central Bay contain higher PCB levels than those in the northern and southern bay areas (North and South Bay, respectively). Based on the water volume of  $2.2 \times 10^8 \text{ cm}^3$  at a lower water level, it was estimated that approximately 1,000 grams of PCBs are transported out of SDB via tidal exchange annually. This indicates that water column transport driven by tidal exchange is a viable mechanism reducing PCB contamination within SDB.

## INTRODUCTION

San Diego Bay (SDB), located in southern California (Figure 1), is one of the most urbanized coastal regions in the United States and is listed as an impaired water body by the State of California under Section 303(d) of the Federal Clean Water Act (California State Water Resources Control Board 1998). Heavy commercial and military shipping activities and surface runoff draining densely populated areas have resulted in highly elevated levels of a number of organic contaminants and trace metals in sediments and biota (Mearns *et al.* 1991, McCain *et al.* 1992, Fairey *et al.* 1998b). Particularly, concentrated polychlorinated biphenyls (PCBs) were found

in sediments along the northeastern side of the Central Bay (McCain *et al.* 1992, Fairey *et al.* 1998a). Several large-scale monitoring and research programs have documented the spatial and/or temporal (to a lesser extent) distributions of sediment and biota contamination and toxicity (McCain *et al.* 1992, San Diego Bay Interagency Water Quality Panel 1994, Fairey *et al.* 1998a, Noblet *et al.* 2002). Despite a fair amount of effort to monitor contaminant distributions in SDB, no studies have been directed toward measurements of water column contamination.

The California Ocean Plan specifies discharge limits for a large number of chemicals in the receiving waters of California (California State Water Resources Control Board 1997). However, very few studies have directly measured water contaminants in coastal waters because of technical difficulties in the measurement of ultra-low-level chemical contaminants. A previous study found that both DDT and



**Figure 1. Sampling locations in San Diego Bay. Stations 1, 2, and 3 are located in North Bay; Stations 4, 5, 6, and 7 in Central Bay; and Stations 8 and 9 in South Bay.**

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PCB concentrations in the water column of the Palos Verdes Shelf greatly exceeded the California Ocean Plan discharge limits (Zeng *et al.* 1999). Because dilution may be limited in a semi-closed embayment such as SDB, water column contamination may be more persistent compared to an open environment. In addition, the water column is a critical component for redistribution and dispersal of historically discharged chemical contaminants.

Another important issue is whether contaminated sediments in SDB have become a relatively important source of contamination compared to other historically known sources. As a consequence, these sediments may have continued to recycle historically deposited contaminants to the surrounding environments. This hypothesis was proposed previously as a probable environmental process in the Southern California Bight (Zeng and Venkatesan 1999; Zeng and Tran 2002).

To address these issues, we measured the concentrations of PCBs in the water column of SDB with an *in situ* water sampling system capable of processing large volumes of water (Zeng *et al.* 1999). The sampling strategy allowed detection of ultra-low levels of chemical contaminants so that a meaningful comparison with the discharge limits specified in the California Ocean Plan could be made. The present study is the first of its kind to measure ultra-low levels of organic contaminants in the waters of SDB.

## METHODS

### Field Sampling

Sampling was conducted in summer 1999 (from June 15 to July 1) and winter 2000 (from January 31 to February 29) at nine locations within SDB (Figure 1). The selection of the sampling sites was intended to spatially sample the entire Bay to assess the water column contamination of PCBs, and was not based on past records of sediment PCB contamination. Water column samples were collected with an *in situ* pumping and filtering method (Zeng *et al.* 1999) at distances of 1.5 and 5 m from the sediment-water interface. Typically, 3-4 d of deployment were required to process up to 2,500 L of water to achieve adequate detection limits. In this study, two to three pumps were used concurrently for each sampling procedure. Each pump housed eight Whatman GF/F glass fiber filters (142 mm in diameter) to retain particles and one Teflon® column packed with XAD-II resins to extract dissolved organics. Upon retrieval

of each pump, the glass fiber filters were placed in glass Petri dishes and sealed. The Petri dishes and Teflon® columns wrapped with aluminum foil were placed in an ice chest during transportation to the laboratory. The Teflon® columns were processed within 24 h after arrival and the glass fiber filters in the Petri dishes were stored at -20°C until further treatment.

### Measurements of Total Suspended Solids

Concentrations of total suspended solids (TSS) were estimated from the particulates retained by the glass fiber filters. Because each sample was composed of eight filters and the same filters were also used for PCB analyses, a subsampling strategy was employed to measure TSS values. A hand-held corer (17.6 mm diameter) was used to take two plugs out of each filter. Two plugs from a blank filter pre-soaked with seawater were also weighed and averaged to provide the blank filter subsample weight. The subsamples were dried at 60°C for more than 4 h and subsequently placed in a desiccator until constant weights were reached. Each subsample weight ( $w_{sub}$ ) from each filter was the sum of two measurements (two plugs) on the difference between each subsampled filter weight and the blank filter subsample weight. The TSS weight ( $W_{TSS}$ ) from each filter was determined as follows:

$$W_{TSS} = \frac{A_{whole}}{A_{subsample}} \times w_{sub}$$

where  $A_{whole}$  is the area of the whole filter and  $A_{subsample}$  is the total area of the two subsamples from the whole filter. The TSS concentration ( $C_{TSS}$ ) was then estimated by

$$C_{TSS} = \frac{\sum_{i=1}^8 W_{TSS}(i)}{V_{water}}$$

where  $V_{water}$  is the volume of water processed by an *in-situ* pump and the number 8 indicates the total number of filters comprising each sample.

### Measurements of Total Organic Carbon and Total Nitrogen

The subsamples taken for the determination of TSS were also used for total organic carbon (TOC) and total nitrogen (TN) measurements. These samples were processed with a procedure described

elsewhere (Zeng and Khan 1994) and analyzed with a Carlo Erba 1108 CHN Elemental Analyzer (Carlo Erba Instruments, Milan, Italy). In addition, blank filter plugs were soaked in seawater and processed along with the particulate-loaded filters to correct for salt-related interferences. The TOC concentration for each sample ( $C_{\text{TOC}}$  in weight %) was an average of 16 measurements from the subsamples minus the background value from the blank filter:

$$C_{\text{TOC}} = \frac{\sum_{i=1}^{16} C_{\text{TOC}}(i)}{16} - \text{TOC\% (blank subsample)}$$

where  $C_{\text{TOC}}(i)$  is the TOC concentration in subsample  $i$ , which was obtained from the TOC weight divided by the corresponding TSS mass for subsample  $i$ . Concentrations of TN were also obtained with the same procedure.

### PCB Analysis

Sample extraction procedures are described by Zeng *et al.* (1999). Target analytes were 27 PCB congeners (18, 28, 29, 44, 50, 52, 66, 77, 87, 101, 104, 105, 118, 126, 128, 138, 153, 154, 170, 180, 187, 188, 195, 200, 206, and 209). Internal standards were PCB 30 and PCB 169, and surrogate standards were tetrachloro-*m*-xylene, PCB 65, and PCB 189.

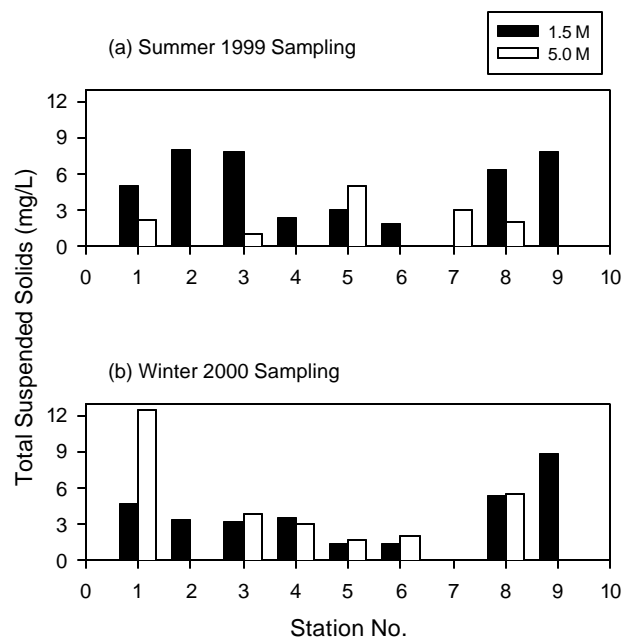
Instrumental analysis was conducted using a Varian 3800 gas chromatograph combined with a Saturn 2000 ion trap mass spectrometer and a Varian 1079 SPI injector (Varian Inc., Walnut Creek, CA). Chromatographic separation was provided by either a 60 m x 0.32 mm i.d. (0.25  $\mu\text{m}$  film thickness) DB-XLB or a 60 m x 0.25 mm i.d. (0.25  $\mu\text{m}$  film thickness) DB-5 column. Helium was used as a carrier gas at a constant flow of 1.3 mL/minute. Injection was performed in split mode with split ratio programmed as following: 1:4 for 0.3 min; off for 2 min; and 1:20 for 20 min. The injector temperature was initiated at 60°C for 0.3 minutes and increased to 310°C at 200 °C/minute. The oven temperature profile was as follows: 60°C for 1 minute; 60-180°C at 15°C/minute; 180-280°C at 2°C/minute; 280-310°C at 5°C/minute; and 310°C for 3 minutes. Chromatograms were processed with a Varian Saturn 2000 Workstation data system. The nominal detection limit was 10 ng for 1 mL of water sample or 1 g of solid sample. The actual detection limit varied with the sample size corrected for the subsamples taken for TSS measurements.

### QA/QC Assessment

The QA/QC measures were similar to those described previously by Zeng *et al.* (1999). Field blank samples, defined as clean water in glass bottles and exposed to the ambient environment during the course of field operation, contained no detectable PCBs. Recoveries of the surrogate standards spiked into the field samples prior to extraction were 75±17% for tetrachloro-*m*-xylene, 85±17% for PCB 65, and 87±10% for PCB 189. The National Institute of Standards and Technology 1493 reference standard containing 20 PCB congeners on the analyte list was analyzed four times during the analyses of field samples and the average recovery of all the PCB congeners from each analysis was 91±5, 91±5, 92±12, and 90±9%, respectively.

### RESULTS

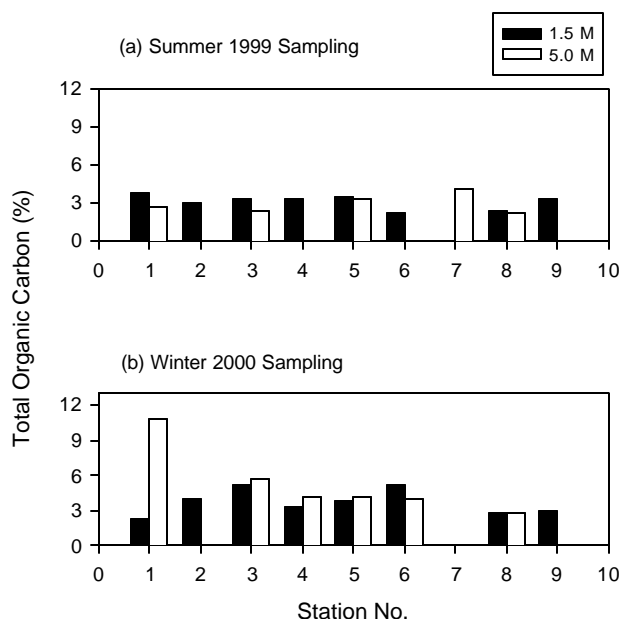
Concentrations of TSS ranged from approximately 1 to 12 mg/L (Figure 2). The median value was 3.0 mg/L for the summer 1999 samples, and 3.5 mg/L for the winter 2000 samples, respectively. Seasonal variation was not apparent. Spatially, TSS concentrations at the 1.5-m depth were slightly higher



**Figure 2. Distribution of total suspended solids (mg/L) in the water column of San Diego Bay sampled during: (a) summer 1999 and (b) winter 2000. Absence of peaks indicated no samples were collected due to technical problems.**

in the North (Stations 1-3) and South (Stations 8-9) bays than in the Central Bay (Stations 4-7), but the pattern for TSS concentrations at the 5-m depth was not clear. In any event, the number of samples was too small to quantify the assessment. An unusually high value of TSS was found in the sample from Station 1 at the 5-m depth during the winter 2000 sampling period. This may have been an artifact due to tampering of the sampling system, as suggested by the displacement (~ 50 m) of the sampling system from its original deployment site that may have caused an inaccurate recording of the sample volume (only 270 L of water were recorded compared to typically 1,000 to 2,000 L). Duplicate samples were collected from Station 5 at the 5-m depth, and the TSS concentrations were 4.2 and 3.3 mg/L, respectively.

Concentrations of TOC in suspended solids ranged from 2 to 10%, but no apparent trend was noticeable spatially and temporally (Figure 3). Variability was not obvious between the samples collected at the 1-m and 5-m depths, except for a large spike at the 5-m depth of Station 1. The median TOC concentration was 3.2% for the summer 1999 samples and 4.0% for the winter 2000 samples, respectively.



**Figure 3. Distribution of total organic carbon (%) in suspended solids collected from the water column of San Diego Bay sampled during: (a) summer 1999 and (b) winter 2000. Absence of peaks indicated no samples were collected due to technical problems.**

The total organic carbon/total nitrogen (C/N) ratio varied in a narrow range,  $6.1 \pm 0.6\%$  for the summer 1999 samples and  $5.9 \pm 0.7\%$  for the winter 2000 samples (not shown).

Total PCB concentrations ranged from nondetectable to 0.42 ng/L (from the 5-m depth of Station 3) (Table 1). In general, higher PCB concentrations occurred in the Central Bay (including Station 3 near the Central Bay) than in the North and South bays. The dissolved phase samples contained higher concentrations of PCBs and a larger number of detectable PCB congeners compared to the particulate samples (volume based). Most PCB congeners detected in the dissolved phase but not in the particulate phase were low-molecular-weight ones and/or had low concentrations. This can be explained by the fact that the distribution of PCBs between the dissolved and particulate phases is dictated by the hydrophobicity of individual PCB congeners and the concentrations of suspended solids. The number of detectable PCB congeners was 14 and 7 for the dissolved and particulate samples of 1999 and 10 and 5 for the dissolved and particulate samples of 2000, respectively (not shown). Duplicate samples collected from Station 5 at the 5-m depth showed similar PCB concentrations (0.21 and 0.27 ng/L), indicating consistency in the sampling and analytical procedures.

A qualitative assessment of the thermodynamics for partitioning of PCB congeners between the particulate and dissolved phases was made from the limited number of detectable PCB congeners. The TOC-normalized equilibrium partition coefficient ( $K_{oc}$ ) is expressed as

$$K_{oc} = \frac{C_{oc}}{C_{w,e}} \quad (1)$$

where  $C_{oc}$  is the TOC-normalized concentration of a specific PCB congener in the particulate phase and  $C_{w,e}$  the concentration of the same compound in the dissolved phase at equilibrium state. If the measured concentration of the compound in the dissolved phase is designated as  $C_w$  (not at equilibrium state), the ratio  $C_{w,e}/C_w$  represents the thermodynamic tendency (or chemical fugacity) for the compound to partition in the dissolved phase (Achman *et al.* 1996). A value of  $C_{w,e}/C_w$  greater than 1 indicates the thermodynamic potential that the compound may move from the particulate phase to the dissolved phase. Rearranging Equation 1 gives:

**Table 1. Concentration distribution of polychlorinated biphenyls in the water column of San Diego Bay. <sup>a</sup>**

Station	1999			2000		
	Dissolved	Particulate	Total	Dissolved	Particulate	Total
(1) 1.5-Meter Depth <sup>b</sup>						
1	0.024	<0.0095	0.024	0.065	0.014	0.079
2	0.08	<0.0097	0.08	0.171	0.07	0.241
3	0.24	<0.016	0.24	0.248	0.124	0.372
4	0.204	<0.019	0.204	0.245	0.135	0.38
5	0.166	0.071	0.237	0.233	0.021	0.254
6	0.246	<0.0089	0.246	0.184	0.028	0.212
8	0.064	<0.0082	0.064	0.14	0.022	0.162
9	0.104	0.016	0.12	0.073	0.005	0.078
(2) 5-Meter Depth <sup>b</sup>						
1	<0.011	<0.011	<0.011	0.054	<0.037	0.054
3	0.127	<0.0072	0.127	0.331	0.088	0.419
4	N/A	N/A	N/A	0.22	<0.011	0.22
5	0.174	0.031	0.205	0.198	0.053	0.251
5 (duplicate)	0.244	0.029	0.273	N/A	N/A	N/A
7	0.152	<0.0082	0.152	0.244	0.017	0.261
8	0.217	<0.012	0.217	0.146	0.019	0.165

<sup>a</sup> All concentrations were in ng/L. N/A = No sample collected.  
<sup>b</sup> Distance from the sediment-water interface.

$$\log \frac{C_{oc}}{C_w} = \log K_{oc} + \log \frac{C_{w,e}}{C_w} \quad (2)$$

Obviously, if  $\log(C_{oc}/C_w)$  is greater than  $\log K_{oc}$ , then  $\log(C_{w,e}/C_w)$  is greater than zero. The  $K_{oc}$  value is not readily available, but can be parameterized to  $K_{ow}$  (octanol-water partition coefficient that has been determined for many chemicals) as  $\log K_{oc} = a \log K_{ow} + b$  with  $a$  and  $b$  being experimentally determined. Substitution of this expression into Eq 2 yields

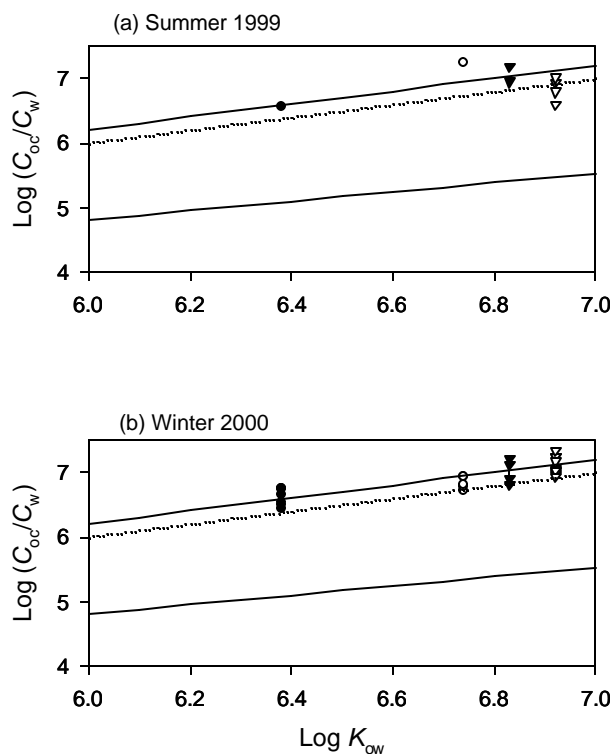
$$\log \frac{C_{oc}}{C_w} = a \log K_{ow} + b + \log \frac{C_{w,e}}{C_w} \quad (3)$$

Karichhoff *et al.* (1979) and Schwarzenbach and Westall (1981) proposed two sets of  $a$  and  $b$ , representing the high and low ends of  $K_{oc}$  values measured experimentally. Karichhoff *et al.* (1979) obtained  $a = 1.0$  and  $b = 0.21$ , while Schwarzenbach and Westall (1981) obtained  $a = 0.72$  and  $b = 0.49$ . Eq 3 indicates that  $\log(C_{oc}/C_w)$  values from all detectable PCB

congeners with varying  $K_{ow}$ 's can be compared to the plot  $a \log K_{ow} + b$  vs.  $\log K_{ow}$  to assess the thermodynamic potentials for partitioning between the particulate phase and the dissolved phase.

Figure 4 shows the values of  $\log(C_{oc}/C_w)$  obtained from PCB 101, PCB 118, PCB 138, and PCB 153, which were detected in both the dissolved and particulate phases for all of the samples. The upper and lower solid lines were obtained from  $a \log K_{ow} + b$  with  $a$  and  $b$  being suggested by Karichhoff *et al.* (1979) and Schwarzenbach and Westall (1981), respectively. The dotted line represents the  $\log K_{oc} - \log K_{ow}$  correlation. All of the data points appeared to fall around the high end of the equilibrium partition range and the  $\log K_{oc} - \log K_{ow}$  correlation line. Because the dissolved phase was operationally defined as all materials (including water) that passed through the 0.7- $\mu$ m pore size filters, truly dissolved PCB concentrations might have been overestimated. However, such effects were unknown and no correction could be made.

Although the present study focused on the analysis of water column samples, sediment cores



**Figure 4. Partition coefficients of PCB 101 (?), 118 (?), 138 (?), and 153 (N) between the suspended solid phase and the dissolved phase in water column samples collected during: (a) summer 1999 and (b) winter 2000. The upper and lower solid lines represent the empirical  $\log K_{oc}$  values proposed by Karichhoff *et al.* (1979) and Schwarzenbach and Westall (1981), respectively. The dotted line is the  $\log K_{ow}$  –  $\log K_{oc}$  correlation.**

were also collected from each sampling station where water column samples were taken. Concentrations of all individual PCBs were below the detectable limit of approximately 5 ng/g (based on dry sediment weight) in all of the sediment core samples.

## DISCUSSION

The California Ocean Plan establishes water quality objectives for California's oceans as the basis for regulation of wastes discharged into California's coastal waters (California State Water Resources Control Board 1997). These objectives apply to point and nonpoint source discharges. The 30-d average discharge limit for PCBs (defined as the sum of Aroclors) is 0.019 ng/L. The present study, for the first time, measured the water column PCB concentrations in SDB, which allowed a direct assessment of

the water quality in SDB based on the California Ocean Plan's objectives. The PCB concentrations in all but one of the samples (Station 1 at the 5-m depth) collected throughout the Bay were higher than the discharge limit (Table 1). The extent of exceedance would have been greater if more PCB congeners had included for measurements and/or the analytical sensitivity been increased so that more PCB congeners were detected. Nevertheless, this exceedance indicates that PCB contamination remains a significant problem for the beneficial uses of SDB.

Existing data indicate that the water PCB concentrations in SDB (Table 1) are fairly comparable with or lower than those determined in some other closed and semi-closed aquatic systems in the U.S., but higher than those in coastal waters and open oceans around the world. For example, average total PCB concentrations in open waters of Lake Michigan and Lake Superior were 0.47 ng/L (measured in 1991) (Pearson *et al.* 1996). In San Francisco Bay, California, where water PCB concentrations have been monitored almost every year, the latest study conducted in 1999 indicated that the total PCB concentrations ranged from 0.38 to 7.2 ng/L (San Francisco Estuary Institute). Globally, concentrations of total PCBs in the Sea of Japan, a semi-closed "small ocean," at various depths ranged from 0.0003 to 0.005 ng/L in 1995 (Kannan *et al.* 1998). Individual PCB congener concentrations in surface seawater along the coast of Alicante, Spain, were below the detection limits of 0.01 to 0.05 ng/L (Prats *et al.* 1992). A large survey conducted between 1989 and 1990 on various open oceans and seas obtained surface seawater PCB concentrations as high as 0.063 ng/L (North Pacific) (Iwata *et al.* 1993).

One of the probable sources of PCBs detected in the water column of SDB may be contaminated sediments. Historically, concentrated PCBs were largely found in sediments around the Central Bay, particularly near the naval shipyard (near Station 6; Figure 1) and some parts of the northeastern side of the Central Bay (near Stations 3 and 4; Figure 1) (Mearns *et al.* 1991; McCain *et al.* 1992, Fairey *et al.* 1998a, Chadwick *et al.* 1999). The distribution of water column PCB concentrations (with higher concentrations in the Central Bay than in the North and South bays; Table 1) appeared to agree with the previously measured sediment PCB distribution. This agreement was further corroborated by the lower concentrations of TSS in the Central Bay relative to those in the North and South bay (Figure 2). If PCBs

were significantly derived from the North and South bays, the higher TSS concentrations found in these regions would have provoked higher PCB concentrations in the water column because of the high hydrophobicity of PCB congeners.

The fact that individual PCB congeners were all below detectable levels in sediments simply indicated that the water column PCBs may have been derived from sediments in areas not sampled in the present study. A nearly equilibrium distribution of PCB congeners between the particulate and dissolved phases (Figure 4) also suggested a non-localized source of PCBs was present in the sampling locations. PCBs in freshly resuspended particles would tend to partition into the water phase if the sediments contained enriched amounts of PCBs, because the overlying water was presumably depleted in PCBs relative to the suspended particles.

It is worthwhile to note that sediments in one particular area (Convair Lagoon) nearshore off the San Diego International Airport (close to Station 3; Figure 1) contained highly elevated levels of PCBs. About half of the area (5.7 out of 10 acres) was capped in 1998 with crushed rock and sand. Recent investigations, however, found PCBs on top of the cap and near a storm drain (San Diego Regional Water Quality Control Board). This finding has compromised our efforts to relate the water PCB contamination to that in sediments. Although more studies are needed to confirm the sources of the "new" PCBs, presently surface runoff may not be completely ruled out as a source of fresh PCB inputs into SDB. On the other hand, sediments in other known contaminated areas (e.g., near the naval shipyard) are expected to contribute PCBs to the water column. Calculations based on a recent survey conducted in 1998 (Noblet *et al.* 2002) indicated that the area-weighted mean PCB concentration in SDB sediments (46 samples collected and analyzed) was 6.9 ng/g (dry-weight based) with a range from below 1 to 124 ng/g. The sediment PCB concentrations (from below 1 to 124 ng/g) occurring near the naval shipyard were lower than those obtained from a previous survey conducted between 1992 and 1994 (Fahey *et al.* 1998a). This apparently decreasing trend of PCB concentrations may be indicative of redistribution of PCBs from sediment to water and transport of PCBs out of SDB.

The widespread presence of PCBs in the water column allowed an estimate of losses of PCBs due to water exchange. The average and median concen-

tration of total PCBs was approximately 0.2 ng/L (Table 1). This concentration was used to represent the average concentration of PCBs in the water column of SDB. The water volume in SDB for a lower water level is roughly  $2.2 \times 10^8$  m<sup>3</sup> (Chadwick *et al.* 1999). This yields a total PCB mass of 44 g in the water body of SDB at any given time. A model calculation based on a tidal amplitude of 0.8 m produced a time of 8.1 d for a 50% exchange of water (San Diego Bay Interagency Water Quality Panel). Assuming PCBs in the water column can be exchanged completely with water, an annual loss of PCBs due to tidal exchange is approximately 1,000 g. Since we used the water volume at a lower water level, this number might represent the low end of the actual losses, assuming other variables were correct.

If the loss of the total PCB mass is a result of the reduction of PCBs in the top 1-cm sediments within the whole Bay with an area of  $4.1 \times 10^7$  m<sup>2</sup> (Chadwick *et al.* 1999), the annual loss of sediment PCB concentration would be 2.5 ng/mL (volume of wet sediment) or 2.5 ng/g (dry sediment weight) if the moisture content is offset by the dry sediment density (e.g., the moisture content is 50% and the dry sediment density 2 g/mL). Apparently, only a small portion of the sediments within SDB contributed significant amounts of PCBs to the water column. The actual loss in sediment PCB concentration would be much greater than 2.5 ng/g for "hot spots." For example, if sediments from only 1% of the area contributed to the water column PCBs, the decrease in PCB sediment concentrations would be 250 ng/g per year. Nevertheless, tidal exchange may be a viable mechanism for reducing PCB contamination within SDB.

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