

Adsorption and desorption of tributyltin in sediments of San Diego Bay and Pearl Harbor

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Adsorption and desorption of butyltin compounds from sediment under simulated estuarine conditions depends upon the characteristics of the sediment including grain size distribution, percentage of organic carbon, clay mineralogy and aqueous butyltin concentration in the overlying water column. Sediments from Pearl Harbor, Hawaii, USA, primarily consisting of calcium carbonate mud and 18–28% organic carbon by weight, have generally abundant adsorption sites and display tributyltin partition coefficients (K_p) ranging from 1000 to 5000 $\mu\text{g kg}^{-1}$ per $\mu\text{g dm}^{-3}$. Adsorption and desorption of butyltin from San Diego Bay, California, USA, sediments is linearly dependent upon the characteristics of each sediment and the range in K_p values is from approximately 20 to 2500 $\mu\text{g kg}^{-1}$ per $\mu\text{g dm}^{-3}$. Sandy, low-organic carbon sediments have low K_p while fine-grained, relatively organic-rich sediments have high K_p values. Similarly, samples containing significant amounts of high cation exchange capacity (CEC) clay minerals have relatively higher adsorption potentials than those consisting of low CEC minerals.

Keywords: butyltins, adsorption, desorption, sediments, partition coefficient, cation exchange capacity, clay minerals

INTRODUCTION

The tributyltin moiety (TBT) has been entering the marine environment since its inception as an antifouling additive to marine paints. Published TBT sediment–water and particulate–water partition coefficient values

range from 3.4×10^2 to 1.9×10^6 ,^{1–4} with the majority of the values between 1.0×10^3 and 3.0×10^3 ($\mu\text{g kg}^{-1}$ TBT in sediment per $\mu\text{g dm}^{-3}$ TBT in water).

Since legislative action has led to a lowering of butyltin concentrations in some studied estuarine waters,⁵ there is the potential for sediment to desorb these compounds. Therefore, there exists a need to better understand the factors that control adsorption and desorption of butyltin compounds in order to predict the fate and distribution of these compounds.

This paper describes the influences of organic carbon, grain size distribution and mineralogy on potential adsorption and desorption of butyltin compounds in sediments from San Diego Bay and Pearl Harbor (USA).

EXPERIMENTAL

Collection and preparation

Bottom water samples were collected in 1-dm³ polycarbonate bottles, placed in ice containers, and frozen within 24 h of collection. Sediments were collected in small dredging rigs and placed in polycarbonate bottles. Only the top 6 cm of sediment was collected since it is assumed that the influence of adsorption–desorption equilibrium is most critical at the water–sediment interface. San Diego sediments were collected from 24 February 1986 to 21 January 1988. Pearl Harbor sediments were collected from 9 April 1986 to 10 February 1987. Sample collection sites are presented in Figs 1 and 2.

Sediments were air-dried on watchglasses in a hood, mortar-and-pestled, and stored in glass vials until analyzed or processed. Portions (50 g) allocated for grain size analyses were immersed in 500 cm³ of

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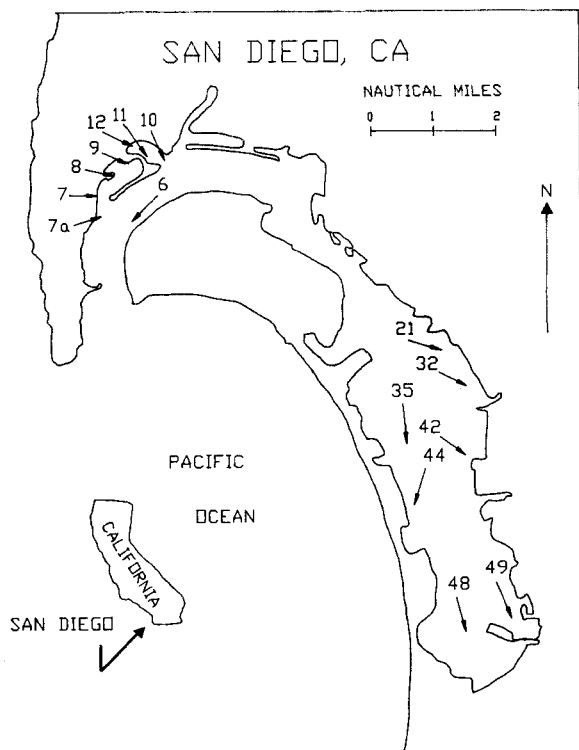


Figure 1 San Diego Bay sample location map.

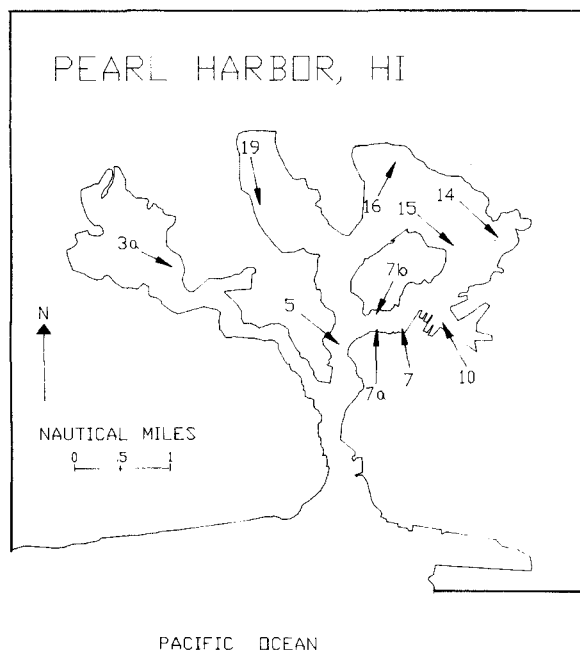


Figure 2 Pearl Harbor sample location map.

deionized water for two weeks (to remove sea salts and deflocculate clay particles), decanted and then air-dried.

Analyses

Water and sediment samples were analyzed for butyltin species (monobutyltin, MBT; dibutyltin, DBT; and tributyltin, TBT) using the hydride derivatization atomic absorption (HD AA) spectrophotometric method⁶ under modified conditions.⁷ Many researchers have treated the polluted sediments with extracting agents and analyzed the extract for organotin.^{7,8} In this case no extracting agent was used. The sediment samples analyzed were directly added to the seawater-filled reaction flask (often referred to as the method of direct addition). The AK_p (apparent partition coefficient) was derived by dividing the original ambient sediment concentration of TBT (in ng g^{-1}) by the original ambient TBT water concentration (in $\mu\text{g dm}^{-3}$).

Loss on ignition (LOI or approximate organic carbon content) was determined by combustion analyses. Approximately 3 g of air-dried sediment was heated to 550°C for 3 h and reweighed.

Grain size distribution was determined by the use of a computerized pipet technique devised by Tom Rockwell of San Diego State University, USA (SDSU). This technique operates by separating sediment fractions as they settle out of a water/sediment/dispersant slurry.⁸ Sand size particles range from 2 mm to $63 \mu\text{m}$ in diameter, silt ranges from $63 \mu\text{m}$ to $2 \mu\text{m}$ in diameter, and clay particles are less than $2 \mu\text{m}$. Clay size fractions were X-rayed on a Diano XRD-8000 diffractometer to determine their mineralogy.

Partition experiments

In the TBT adsorption experiment, 1 g of each sediment was added to 1 dm^3 of seawater containing 118 ng dm^{-3} of TBTCl . Each sediment/water sample (as well as a control containing no sediment) was shaken for 24 h in a lighted room. Water aliquots (decanted from the slurry after settling for at least 2 h) were analyzed for concentrations of MBT, DBT, and TBT adsorbed or desorbed by the sediment. The 118 ng dm^{-3} inoculation concentration in the water was used because bottom water concentrations found in highly polluted regions commonly average around

100 ng dm⁻³ TBT.¹⁰ Butyltin degradation (biological, physiochemical, or photolytic) may have occurred during the adsorption experiment but over the time period it is assumed that all the TBTCl lost from the water was absorbed by the sediment.

Since each sediment sample is inherently unique in terms of exposure regime and proximity to source region, 'T(118)' (original sediment TBT concentration plus or minus amount adsorbed or desorbed by the sediment upon exposure to a 118 ng dm⁻³ TBT water concentration) will serve as a relative measurement of adsorption potential. The experimental partition coefficient, $K_p(118)$, was derived by dividing $T(118)$ by the resultant TBT water concentration (118 ng dm⁻³ plus or minus the amount adsorbed or desorbed by the sediment).

The TBT desorption experiment was conducted identically to the adsorption experiment with the exception that only freash seawater (with no detectable butyltin) was added to each gram of sediment. This phase of the study was run in order to observe the

behavior of the butyltin species when exposed to non-contaminated seawater as a consequence of tidal flushing and/or elimination or reduction of point sources of TBT.

Data

Table 1 lists values for environmentally impacted TBT concentrations (in ng g⁻¹), percentage LOI, experimental TBT adsorption concentrations in ng g⁻¹ (for a 118 ng dm⁻³ TBT inoculation concentration), apparent partition coefficient (AK_p) in units of $\mu\text{g kg}^{-1}$ TBT in sediment per $\mu\text{g dm}^{-3}$ TBT in water, and experimental partition coefficient [$K_p(118)$] in units of $\mu\text{g kg}^{-1}$ TBT in sediment per $\mu\text{g dm}^{-3}$ TBT in water. Table 2 lists values for the same sediments for environmentally impacted TBT, DBT, and MBT concentrations (in ng g⁻¹), experimental adsorption values for each species, and experimental desorption values for MBT and DBT [$M(\text{Des})$ and $D(\text{Des})$, located under the 118 ppt (i.e. ng dm⁻³) TBT

Table 1 Sediment data

Sample	TBT(ng g ⁻¹)	LOI(%)	T(118)	AK_p	$K_p(118)$
SDM-06	<2	1.6	2	400	17
SDM-07	5	4.6	30	123	322
SDM-07A	6	9.8	51	127	699
SDM-08	21	9.2	56	93	675
SDM-09	60	18.1	118	270	1967
SDM-10	66	8.6	95	2060	1070
SDM-11	242	12.5	266	3230	2560
SDM-12	164	5.3	*54	2190	237
SDM-21	26	10.0	59	1160	694
SDM-32	17	10.2	79	2040	1410
SDM-35	3	3.3	33	1610	375
SDM-42	4	6.1	40	429	488
SDM-44	<2	3.3	21	167	211
SDM-48	<2	6.9	26	267	280
SDM-49	4	10.5	62	32	1090
PHM-03A	12	21.1	101	6670	3480
PHM-05	8	18.0	96	3270	3200
PHM-07	402	22.6	407	55100	3600
PHM-07A	121	21.5	168	17800	2370
PHM-07B	5	19.2	94	2270	3240
PHM-10	2830	28.3	*1870	292000	1720
PHM-14	3	27.2	94	357	3140
PHM-15	3	26.1	99	1940	4500
PHM-16	4	19.9	81	1010	1980
PHM-19	5	22.3	94	4370	3240

TBT, original sediment TBT concentration; LOI, loss on ignition; T(118), TBT in sediment after exposure to a water concentration of 118 ng dm⁻³ (ppt); AK_p , apparent partition coefficient (in $\mu\text{g kg}^{-1}/\mu\text{g dm}^{-3}$); $K_p(118)$, experimental partition coefficient. * Desorption of TBT occurred during the adsorption experiment.

Table 2 Sediment data (continued)

Sample	TBT	MBT	DBT	118 ppt TBT		0 ppt TBT		
				<i>M</i> (Des)	<i>D</i> (Des)	<i>M</i> (Des)	<i>D</i> (Des)	<i>T</i> (Des)
SDM-06	<2	<2	<2	<2	<2	<2	<2	<2
SDM-07	5	36	4	10	17	18	10	8
SDM-07A	6	34	8	11	20	11	9	5
SDM-08	21	8	21	5	2	21	14	12
SDM-09	60	103	94	36	139	44	176	26
SDM-10	66	104	93	12	140	50	225	40
SDM-11	242	185	265	40	301	95	489	74
SDM-12	164	111	108	52	184	94	355	149
SDM-21	26	22	29	8	20	4	34	14
SDM-32	17	44	31	2	23	14	53	7
SDM-35	3	11	8	3	5	3	17	2
SDM-42	4	14	5	3	7	3	22	4
SDM-44	<2	17	6	2	1	5	<2	<2
SDM-48	<2	8	4	2	1	4	6	<2
SDM-49	4	9	5	1	1	7	16	3
PHM-03A	12	19	15	9	3	14	16	<2
PHM-05	8	61	28	<2	8	5	32	3
PHM-07	402	108	84	77	257	64	439	49
PHM-07A	121	57	73	15	152	34	193	19
PHM-07B	5	14	11	4	3	3	33	4
PHM-10	2830	533	367	537	1500	831	1860	2780
PHM-14	3	7	6	5	3	6	10	5
PHM-15	3	5	4	<2	<2	7	8	3
PHM-16	4	104	85	8	5	11	5	4
PHM-19	5	211	65	14	6	22	7	<2

TBT, DBT, and MBT are the original sediment TBT, DBT, and MBT concentrations, respectively (in ng g⁻¹); *M*(Des), *D*(Des), and *T*(Des) are concentrations of MBT, DMT, and TBT desorbed (in ng g⁻¹); 118 ppt and 0 ppt refer to water TBT concentrations during the experiments.

heading] for a 118 ng dm⁻³ TBT inoculation concentration. Values obtained for *M*(Des) and *D*(Des), under the 118 ppt heading, represent the amounts of MBT and DBT desorbed during the adsorption experiment.

Values for the concentration of TBT in water (in ng dm⁻³) and depths of collection are presented in Table 3. These values represent bottom (less than 1 m off the sediment/water interface) water concentrations. Water samples were collected and analyzed in triplicate.

Figures 3 and 4 display butyltin concentrations measured in sediment samples of the two regions studied. As expected, highest butyltin concentrations were found in sediments adjacent to dry-dock facilities and regions of relatively dense shipboard activity. Sediments in these regions could prove to be TBT sources upon reduction of TBT concentrations in the

water column following clean-up activities, tidal influences, or heavy precipitation.

Results from sediment grain size analyses are graphically presented in Figs 5 and 6. Relative clay mineralogy can be found in Table 4. No clay minerals were identified in the clay size fractions from Pearl Harbor sediments.

Expandable clay minerals have higher cation exchange capacities (CEC) than non-expandable clay minerals. A hypothetical sediment consisting of 2% clay-sized particles with 50% of the clay minerals classified as expandable will obviously have a lower CEC than another hypothetical sediment consisting of 50% clay-sized particles with 50% of the clay minerals classified as expandable. In order to account for this, an Expandable Clay Index (ECI) was derived by multiplying the percentage clay size fraction by the relative percentage of expandable minerals in the clay

Table 3 TBT water concentrations (ng dm^{-3})^a

Sample	Depth (m)	Max. concn ^b	Min. concn ^b	Average
SDM-06	8.0	9	1	5
SDM-07	7.5	36	30	34
SDM-07A	7.5	36	30	34
SDM-08	5.5	337	133	222
SDM-09	5.5	337	133	222
SDM-10	4.5	56	17	32
SDM-11	5.0	103	43	75
SDM-12	5.0	103	43	75
SDM-21	11.0	26	18	22
SDM-32	11.5	12	6	8
SDM-35	4.0	7	0	2
SDM-42	11.0	12	5	9
SDM-44	4.0	11	3	6
SDM-48	2.5	5	3	4
SDM-49	2.5	43	23	31
PHM-03A	4.0	3	1	2
PHM-05	15.0	3	2	2
PHM-07	15.5	11	3	7
PHM-07A	12.5	11	4	7
PHM-07B	15.5	3	1	2
PHM-10	12.0	12	7	10
PHM-14	6.0	7	5	6
PHM-15	13.0	2	1	2
PHM-16	4.5	5	3	4
PHM-19	6.0	2	1	1

^aValues obtained from P F Seligman, personal communication, 1988 (see text for sample collection dates). ^bFrom replicate analyses.

mineral fraction of the clay-sized fraction (or %E). One must use caution when doing so, since there is no definitive way to quantify this %E value, which depends upon the relative clay mineral portion of the clay-sized fraction and ignores the non-clay mineral portion altogether.

RESULTS AND DISCUSSION

Theoretically, the adsorption and desorption of TBT is a function of the water concentration and properties of the sediment, in particular the clay ($<2\ \mu\text{m}$) and silt plus clay ($<63\ \mu\text{m}$) fractions, the amount of organic carbon present (reflected in the LOI value), and the type of clay minerals present in the $<2\ \mu\text{m}$ size fraction. Percentage $<2\ \mu\text{m}$ size particles and percentage LOI are closely related, since not only do clay particles and organic matter accumulate in low-

energy environments, but they also adsorb on to each other. A significant portion of the percentage $<2\ \mu\text{m}$ size fraction may in fact consist of organic particles.

When searching for trends in TBT partitioning within each harbor, it may be desirable to exclude results obtained for sediments collected at or near dry-dock facilities and high input areas because of biases introduced by very high TBT water concentrations. Also, paint chips (coated with TBT) are often present in these sediments. The HD AA detector can not distinguish between a paint chip coated with TBT and TBT which sorbed to a particle from the dissolved state.

When one compares AK_p values with the TBT water concentrations measured at each station, it appears that many of the sediments are potential TBT sinks, provided literature partition coefficient values are accurate. Relatively high AK_p values (higher than 10 000) are often associated with paint chips in the sediment. Partition coefficient values near 3000 are probably close to equilibrium with respect to TBT

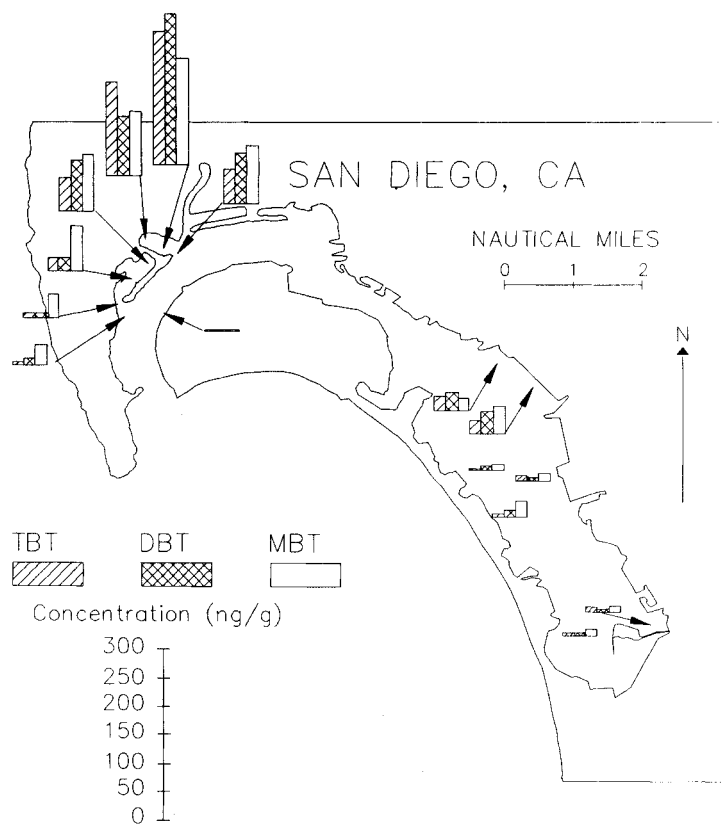


Figure 3 Butyltin species in San Diego Bay sediments.

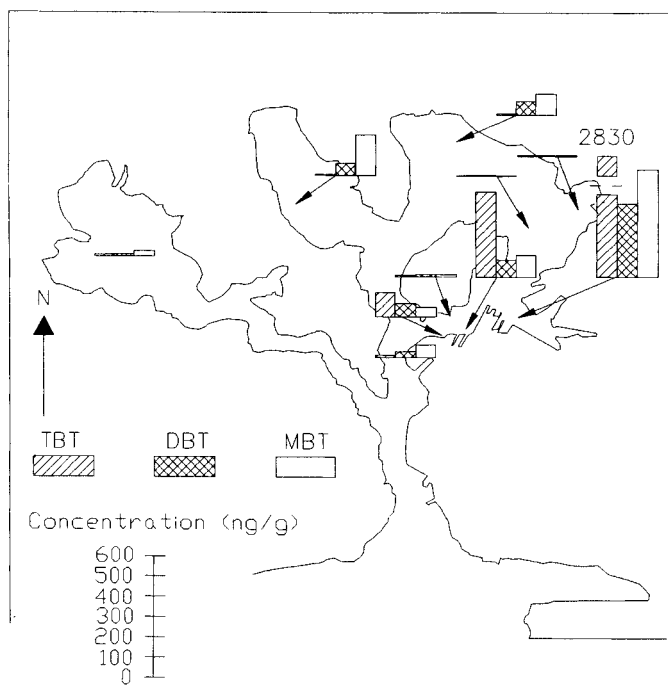


Figure 4 Butyltin species in Pearl Harbor sediments.

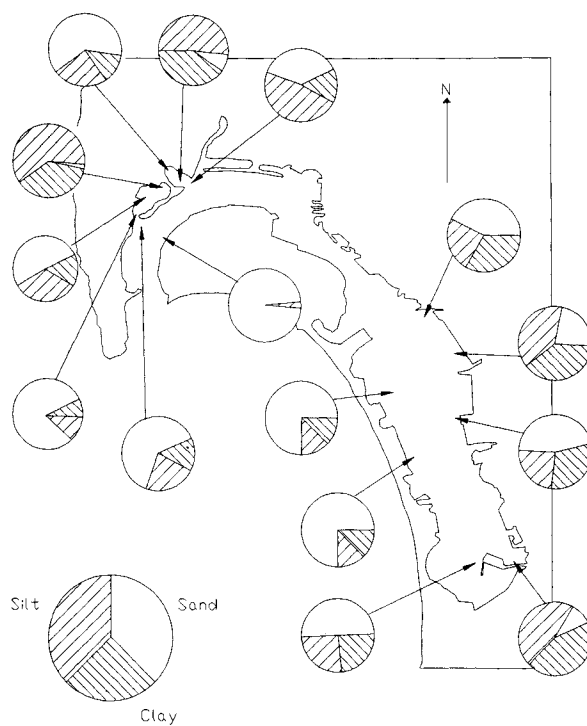


Figure 5 Grain size distribution for San Diego Bay sediments.

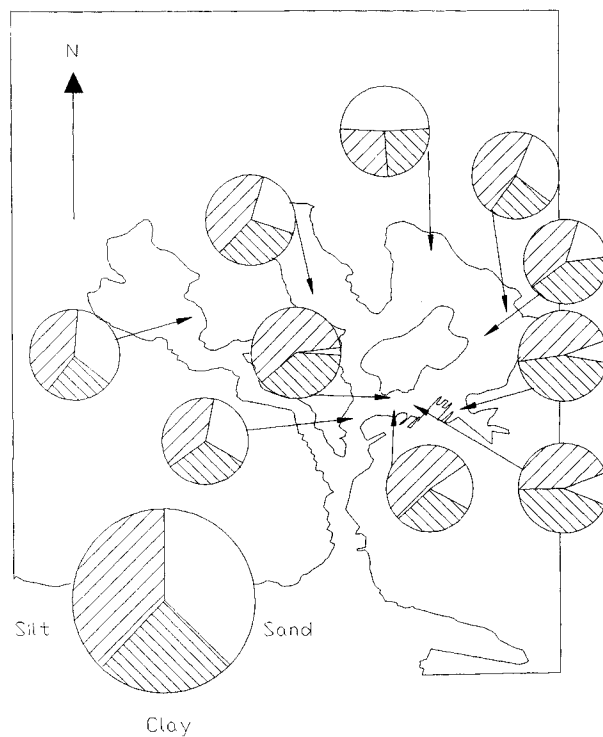


Figure 6 Grain size distribution for Pearl Harbor sediments.

Table 4 Clay mineralogy data^a

Sample	K (%)	C (%)	E (%)	I (%)	ECI
SDM-06	0	0	27	73	16
SDM-07	0	13	40	47	352
SDM-07A	0	13	37	50	577
SDM-08	0	14	38	48	749
SDM-09	0	3	50	44	1520
SDM-10	3	11	41	45	812
SDM-11	0	12	37	50	1454
SDM-12	10	0	32	60	512
SDM-21	0	11	47	42	1391
SDM-32	0	13	41	46	1472
SDM-35	11	2	41	47	533
SDM-42	1	11	46	42	1417
SDM-44	0	10	49	41	676
SDM-48	0	13	45	42	1058
SDM-49	14	1	41	44	1808

Abbreviations: K, kaolinite; C, chlorite; E, expandable minerals; I, illite; ECI, Expandable Clay Index.

^aExpandable minerals were not identified in Pearl Harbor sediments.

partitioning if one disregards paint chip particle inputs.³

Although $K_p(118)$ values are scattered, many fall within the range of 1000 to 4000, suggesting that equilibrium was approached during the adsorption experiment. Averages and standard deviations for $K_p(118)$ values of the two harbors are as follows: for San Diego Bay, average $K_p(118)$ is 806 with a standard deviation of 711; for Pearl Harbor, average $K_p(118)$ is 3047 with a standard deviation of 821. If one accepts literature values for K_p at equilibrium fairly rapidly (within 24–30 hours). This is a significant factor and suggests that these sediments can play a major role in the complex fate of butyltin compounds, since they apparently rapidly respond to changes in water TBT concentration.^{1,10}

Many of the sediment samples from these harbors display a $T(118)$ value of 100 ng g^{-1} . This may represent an equilibrium concentration for a 'typical' 1-g sediment sample in equilibrium with a TBT water concentration of 118 ng dm^{-3} (ppt). Every sample that has a $T(118)$ value close to 100 ng g^{-1} also has a $K_p(118)$ value between 1000 and 5000, suggesting equilibrium was attained during the adsorption experiment. For samples which do not reach this value, sediment characteristics must be such that sufficient available adsorption sites are not present.

In an attempt to identify dependent variables, only

diagrams believed to be pertinent will be presented and discussed. A more complete treatment of the data can be found elsewhere.¹⁰ Notice that some samples were excluded from the variation diagrams for reasons presented in the captions of the appropriate figures.

Percentage LOI versus TBT adsorption capacity

In San Diego Bay sediments, TBT adsorption is directly correlated to percentage LOI (Fig. 7). Pearl Harbor sediments, on the contrary, do not adhere to this trend (Fig. 8). With the exception of high input regions (PHM-07, PHM-07A, and PHM-10) it appears that Pearl Harbor sediments possess an upper limit to the amount of TBT held (approximately 100 ng g^{-1}), given a water concentration of 118 ng dm^{-3} .

Pearl Harbor sediments contain very high percentages of LOI (18 to 28%). The abundant organics in these sediments provides each sample with ample binding sites for TBT uptake. This 100 ng g^{-1} TBT value probably represents an equilibrium value for a TBT water concentration of 118 ng dm^{-3} .

Grain size distribution versus TBT adsorption capacity

San Diego Bay sediments display linear relationships between $<63 \mu\text{m}$ size fractions and the capacity to adsorb TBT (Fig. 9), while those from Pearl Harbor do not (Fig. 10). Once again, with one exception, there exists an upper limit to the maximum amount of TBT held by Pearl Harbor sediments.

Expandable Clay Index (ECI) versus TBT adsorption capacity

Figure 11 supports the hypothesis that the adsorptive potential for TBT depends upon the expandable percentage of the clays in each sediment. Different slopes are apparent from the three specified localities in San Diego Bay, reflecting mineralogical variability within the harbor.

Sediments located in high input regions that contain relatively high amounts of expandable minerals (e.g. SDM-11) are TBT sinks. The situation whereby these types of sediments are located in this type of environment (high input) is a worst-case scenario, since the sediment is exposed to significant amounts of TBT, and it may subsequently persist in the region for

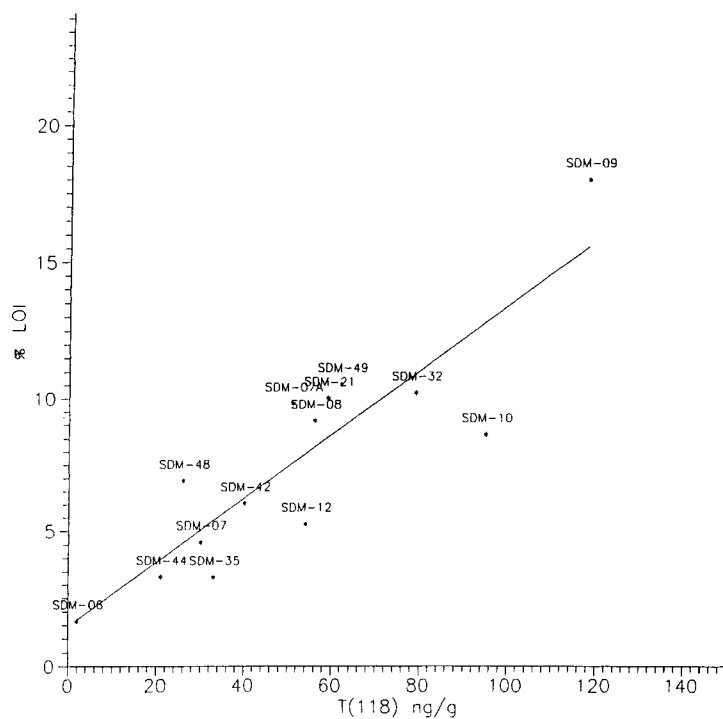


Figure 7 Percentage LOI versus $T(118)$ for San Diego Bay sediments (omitting station SDM-11, which contains paint chips). The correlation coefficient equals 0.88.

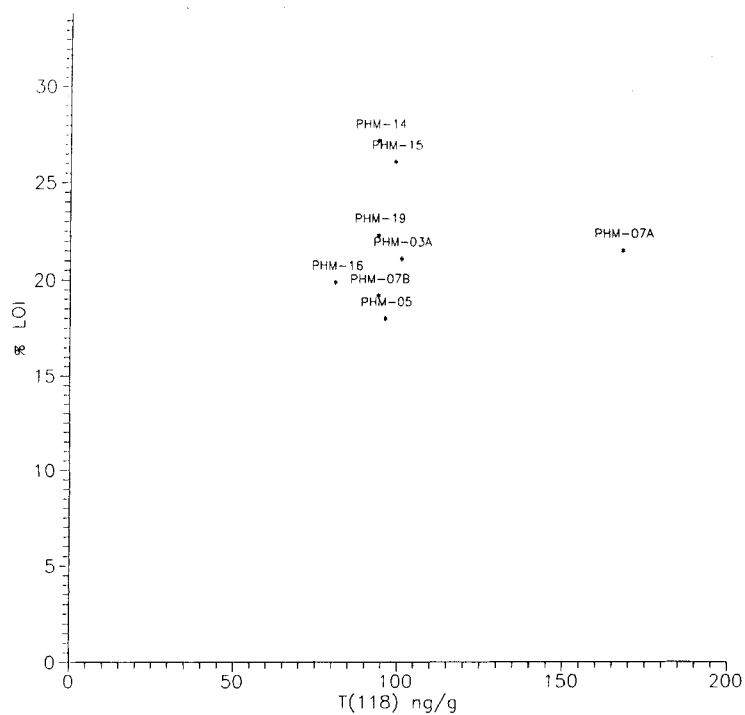


Figure 8 Percentage LOI versus $T(118)$ for Pearl Harbor sediments (omitting stations PHM-07 and PHM-10, which contain paint chips).

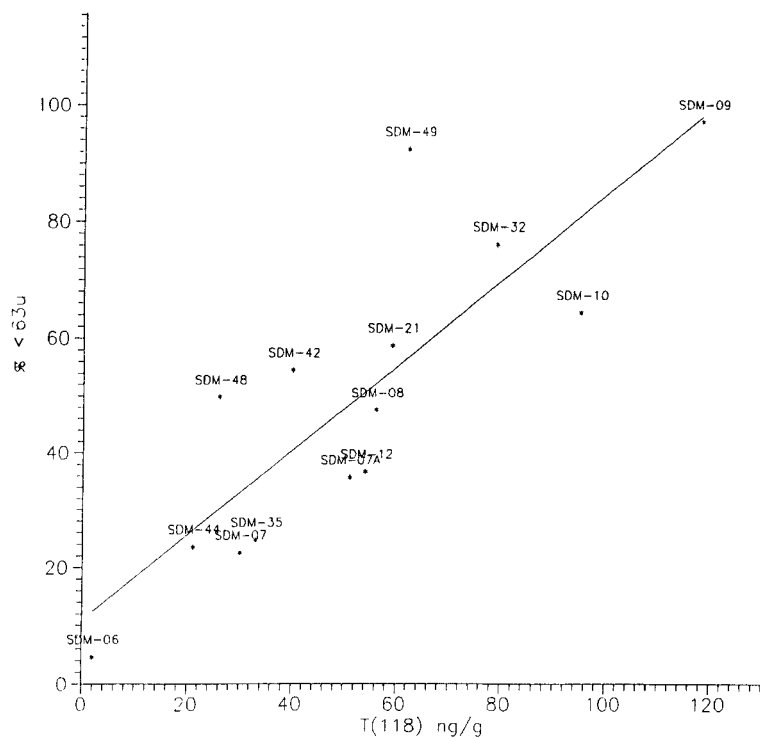


Figure 9 Percentage silt plus clay size particles (<63 μm diameter) versus $T(118)$ for San Diego Bay sediments (omitting station SDM-11, which contains paint chips). The correlation coefficient equals 0.83.

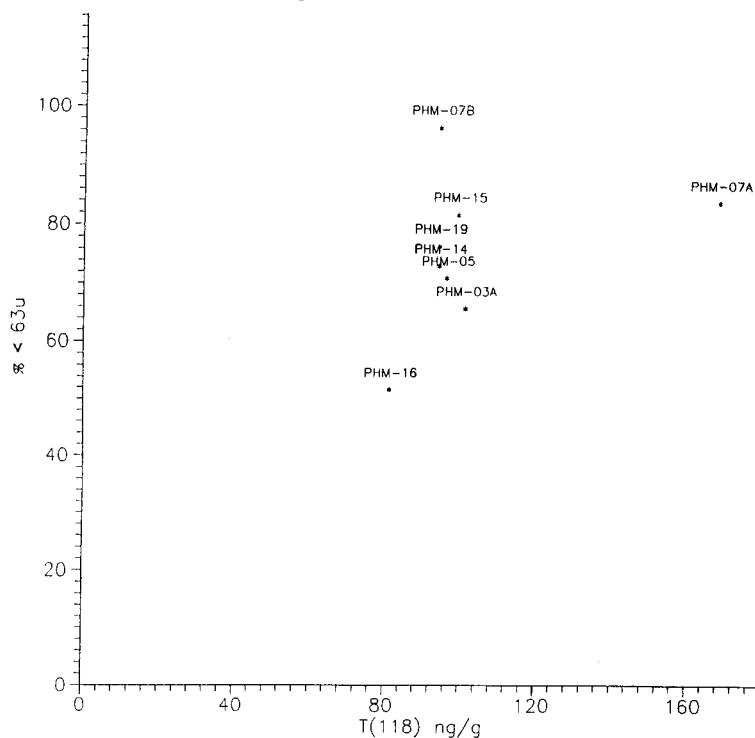


Figure 10 Percentage silt plus clay size particles (<63 μm diameter) versus $T(118)$ for Pearl Harbor sediments (omitting stations PHM-07 and PHM-10, which contain paint chips).

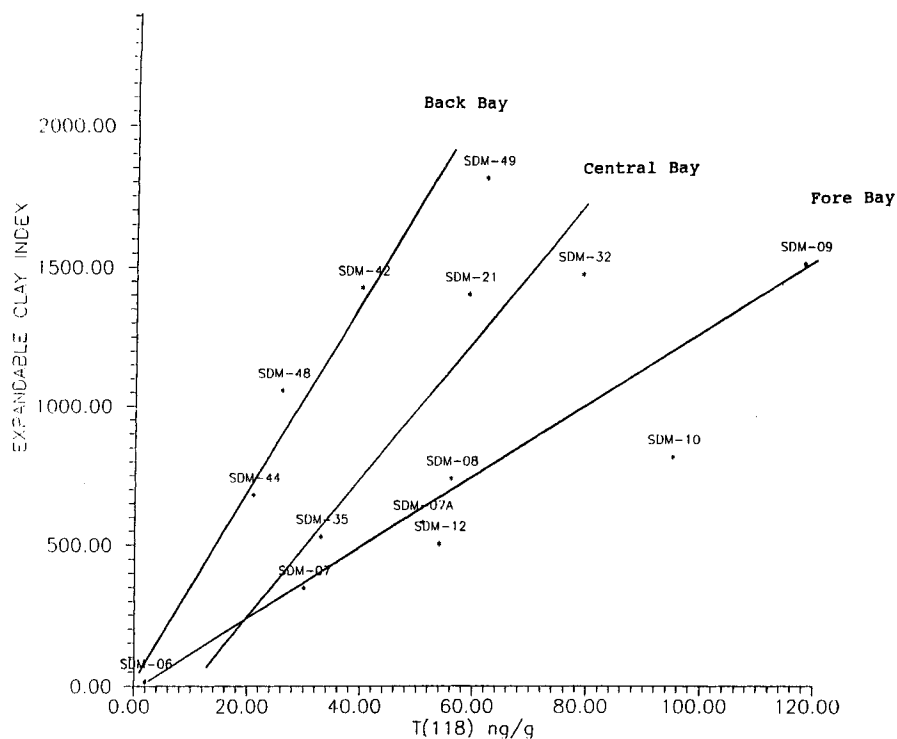


Figure 11 Expandable Clay Index versus $T(118)$ for San Diego Bay sediments (omitting station SDM-11, which contains paint chips). Correlation coefficients are as follows: 0.96 for the back bay; 0.93 for the central bay; and 0.95 for the fore bay.

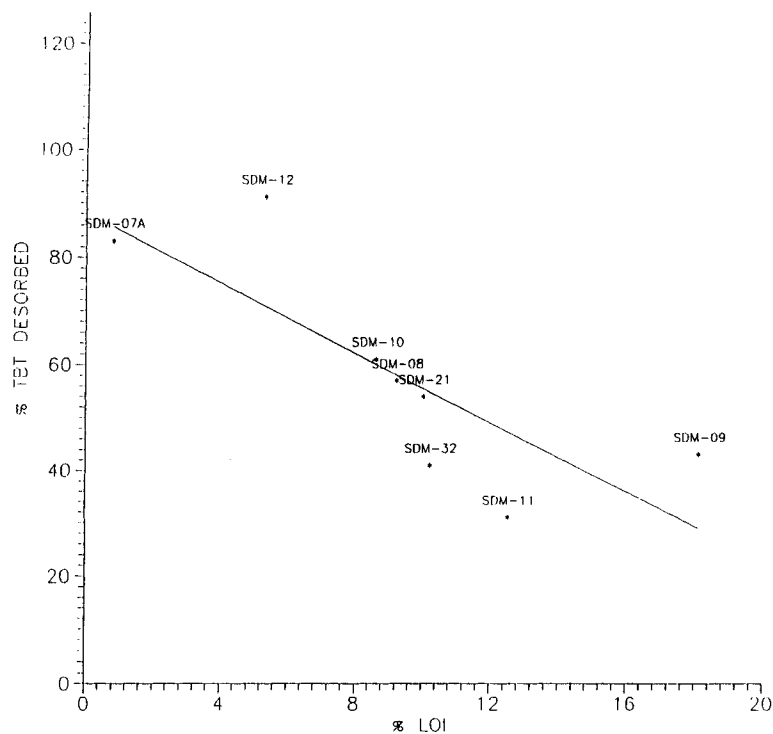


Figure 12 Percentage LOI versus TBT desorbed for San Diego Bay sediments (omitting stations SDM-06, SDM-07, SDM-35, SDM-42, SDM-44, SDM-48 and SDM-49, which contain low initial TBT concentrations). The correlation coefficient equals 0.67.

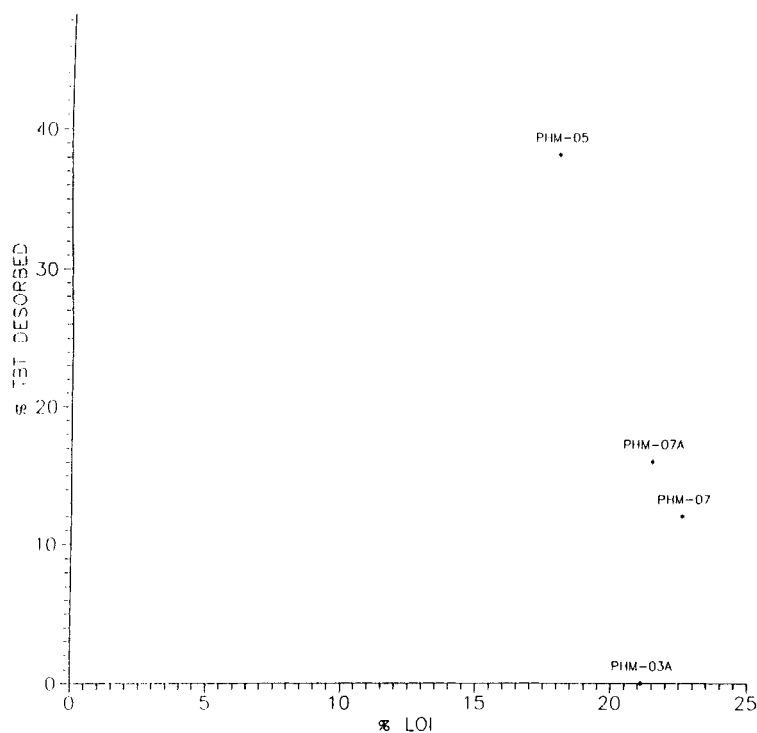


Figure 13 Percentage LOI versus percentage TBT desorbed for Pearl Harbor sediments (omitting stations PHM-07B, PHM-14, PHM-15, PHM-16, and PHM-19, which contain low initial TBT concentrations, and station PHM-10, which displays a mass balance discrepancy).

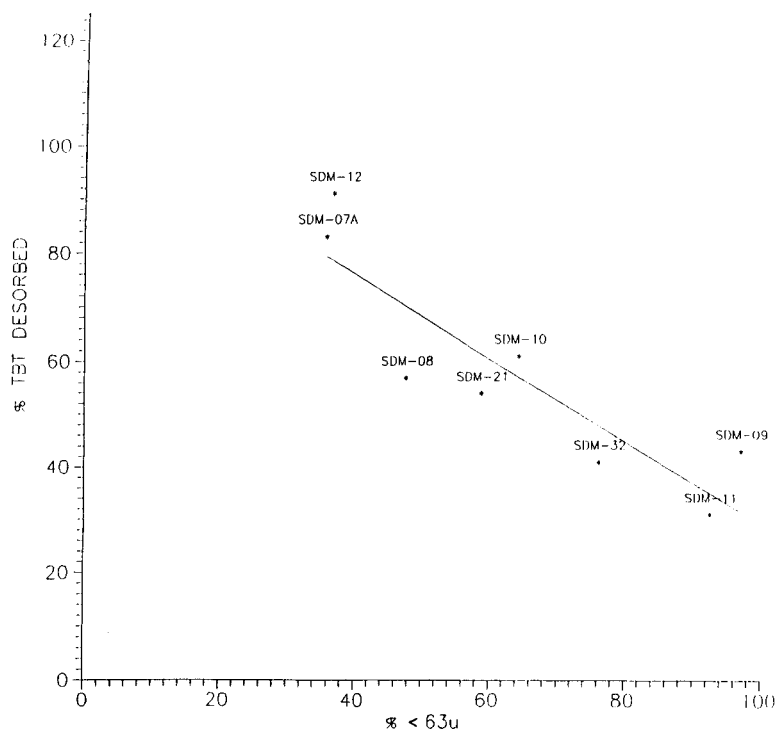


Figure 14 Percentage silt plus clay size particles (<63 μm diameter) versus percentage TBT desorbed for San Diego Bay sediments (omitting stations SDM-06, SDM-07, SDM-35, SDM-42, SDM-44, SDM-48 and SDM-49, which contain low initial TBT concentrations). The correlation coefficient equals 0.89.

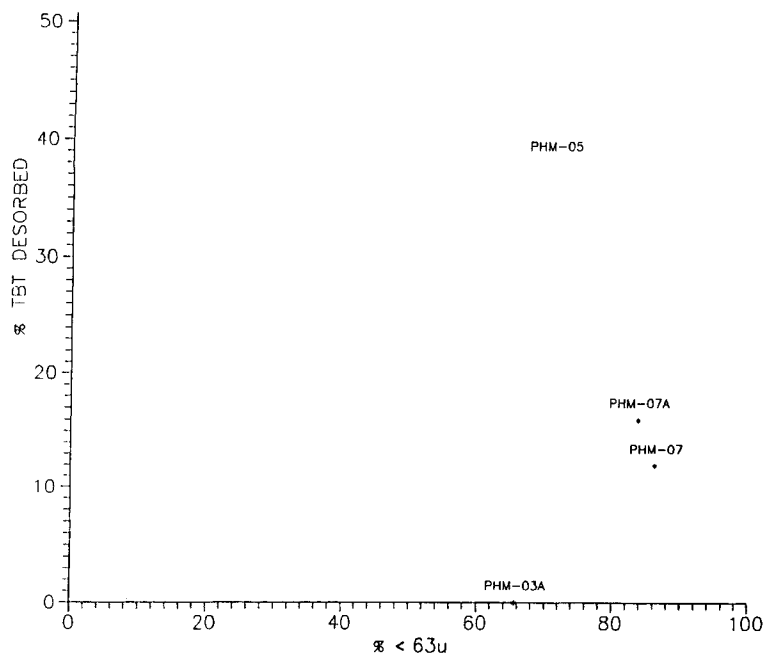


Figure 15 Percentage silt plus clay size particles (< 63 μm diameter) versus percentage TBT desorbed for Pearl Harbor sediments (omitting stations PHM-07B, PHM-14, PHM-15, PHM-16, and PHM-19, which contain low initial TBT concentrations, and station PHM-10, which displays a mass balance discrepancy).

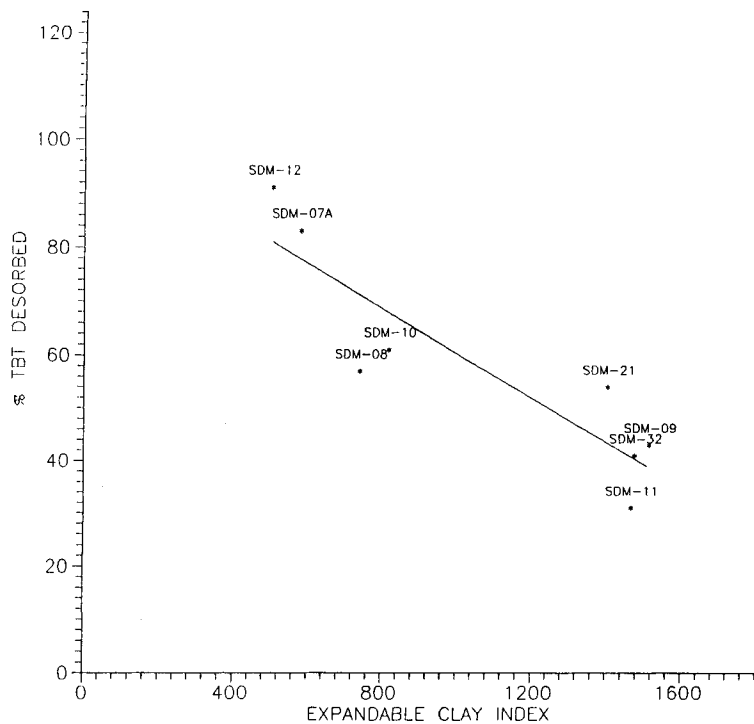


Figure 16 Expandable Clay Index versus percentage TBT desorbed for San Diego Bay sediments (omitting stations SDM-06, SDM-07, SDM-35, SDM-42, SDM-44, SDM-48 and SDM-49, which contain low initial TBT concentrations). The correlation coefficient equals 0.89.

relatively long periods of time. However, others^{11,12} have found TBT degradation half-lives on the order of 4 to 5 months. There is much room for research in this area.

TBT desorption

Figures 12 and 13 illustrate the relationship between percentage LOI and percentage TBT desorbed for the two harbors. When samples containing a concentration less than or equal to 5 ng dm^{-3} (ppt) initial TBT are omitted (because measurements approach detection limits), San Diego Bay sediments display an inversely linear relationship. Pearl Harbor may also adhere to this trend; however, all Pearl Harbor samples analyzed contained sulfide (H_2S) in amounts that may be significant enough to interfere with the organotin measurements.³

Similar relationships hold for each harbor when comparing grain size distribution and mineralogical influences on TBT desorption capacity (Figs 14, 15 and 16). Clearly, percentage LOI, grain size distribution and mineralogy directly influence TBT desorption in San Diego Bay whilst results pertaining to Pearl Harbor sediment desorption are inconclusive.

CONCLUSIONS

Sediment adsorption and desorption characteristics of butyltin compounds may depend upon multiple factors. Analyses relative to sediment characteristics and influence on adsorption and desorption of butyltin compounds in Pearl Harbor and San Diego Bay sediments lead us to four major conclusions:

- (1) Adsorption and desorption of tributyltin depend upon sediment characteristics and water concentration.
- (2) Given enough binding sites (abundant organics, large percentage of silt plus clay, and expandable clay minerals), 100 ng g^{-1} is an approximate equilibrium sediment TBT concentration when exposed to a water concentration of 118 ng dm^{-3} TBT. If

sufficient binding sites are available, TBT partition coefficients range from 1000 to $5000 \text{ } \mu\text{g kg}^{-1}$ per $\text{ } \mu\text{g dm}^{-3}$.

- (3) When sufficient binding sites are not available, desorption and adsorption of butyltin depend directly upon grain size distribution, clay mineralogy and percentage of organic carbon of each sediment sample.
- (4) It is unwise to overgeneralize about partitioning properties of sediments with respect to butyltin. Physical and chemical properties operating in one region may not be applicable to others.

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