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BIOAVAILABILITY OF CHLORINATED HYDROCARBONS AND HEAVY METALS IN SEDIMENTS TO MARINE INVERTEBRATES

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ABSTRACT

Biogeochemical processes affecting bioavailability of chlorinated hydrocarbons and heavy metals in marine sediments are discussed. It is concluded that adsorption-desorption process between sediment and water is the dominating factor controlling bioavailability.

RÉSUMÉ

On discute des procédés biogéochimiques affectant la biodisponibilité des hydrocarbures chlorés et métaux lourds dans les sédiments marins. Nous estimons que l'adsorption-désorption entre le sédiment et l'eau est le facteur dominant contrôlant la biodisponibilité.

The ocean dumping control act of Canada defines hazardous substances in two categories. The first, prohibited substances (Schedule I) includes organohalogen compounds, mercury and cadmium and their compounds. The second, restricted substances (Schedule II) includes several other organic and inorganic compounds.

The regulated limit set for chlorinated hydrocarbons "...that quantity not exceeding 0.01 parts of a concentration shown to be toxic to marine animal and plant sensitive organisms...," is based on a response of the organisms. The limits for mercury and cadmium and their compounds are based on measured concentrations. No such clear guidelines are set for Schedule II substances.

ORGANOCHLORINE COMPOUNDS

Determination of the acute lethality of a series of chlorinated hydrocarbons in sea water to a series of marine invertebrates is a relatively routine matter. Determinations of chronic or long-term lethality

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and particularly of sub-lethal effects are considerably more difficult. Such information, although important, is of little value for predicting the potential effects of chlorinated hydrocarbons contained in sediments. Knowledge of the bioavailability of such compounds from sediment is required. The bioavailability of the compounds is likely to vary with each sediment because of a number of factors related to the nature of the sediment, including particle size composition and organic carbon content.

Desorption characteristics of the chlorinated hydrocarbons associated with the sediment are important in determining the release of the compounds to sea water. In the natural situation, other factors such as flushing rates, as affected by tidal exchange and water currents will modify the actual levels that remain in the water column at a particular site.

The route of uptake of chlorinated hydrocarbons by marine organisms may be either directly from sediment, by contact with, or by ingestion of sediment or fine suspended particles, or from compounds dissolved in interstitial water or in the water column. Finally, ingestion of "contaminated" food may be an important route. One route of uptake does not necessarily eliminate other routes.

BIOAVAILABILITY FROM SEDIMENTS

In review, Addison (in Swiss et al. 1980) mentioned that the solubility of chlorinated hydrocarbons in water is extremely low in contrast with the large capacity of most sediments to adsorb the compounds. Consequently, measured concentrations of such compounds in water are likely to depend largely on the compounds adsorbed on fine suspended particulate matter. A conclusion was that very little information exists about the availability of chlorinated hydrocarbons adsorbed to sediments to benthic fauna.

ADSORPTION-DESORPTION AND SEDIMENTS

Adsorption of PCB's by sediments occurs rapidly (Hiraizumi et al. 1979; Steen et al. 1979; Muto et al. 1974), but little attention has been given to rates of desorption. Reported rates of desorption range from rapid (Steen et al. 1979) to very slow or negligible (Hiraizumi et al. 1979; Hague et al. 1974). Our work (Wildish et al. 1980) investigated the adsorption and desorption of a representative PCB mixture, Aroclor 1254, by various size fractions of a marine sediment over a range of salinities. The data were fitted to the Freundlich isotherm $S = KC^n$ where S is equilibrium concentration of Aroclor 1254 in sediment (μg/kg wet wt) and C is the equilibrium concentration in water, (μg/L). Provided the exponent is close to unity, the K value is a reasonable approximation of the concentration factor. The K values for adsorption and desorption increased at progressively finer sediment fractions and the K values were considerably smaller for adsorption than for desorption. These differences in K values between adsorption and desorption isotherms indicate that adsorption to sediment is relatively irreversible. As reported previously, it appears that organic carbon content of the sediment fractions may be more important than sediment particle size (Choi and Chan 1976; Steen et al. 1978). Our data for the various size fractions of sediment expressed on an organic carbon basis (μg PCB/kg organic carbon) yielded single isotherms for adsorption, $S = 140,000C^{0.8}$; and desorption, $S = 430,000C^{1.1}$, respectively.

Adsorption of Aroclor 1254 by the silt/clay fraction increased with increasing salinity, the K value based on organic carbon content, ranging from about 27,000 at 0 o/oo salinity to 141,000 at 28 o/oo. Our data on desorption in relation to salinity was inconclusive, 1 of 3 tests indicating progressively less desorption with increasing salinty. Huang (1974) reported that salinity had little effect on desorption of several chlorinated hydrocarbons.

UPTAKE OF PCB FROM SEDIMENT BY MARINE INVERTEBRATES

The extent to which PCB's are accumulated from sediments by benthic fauna is of considerable importance. A high degree of accumulation may endanger the fauna directly or the fauna may become a source for accumulation of PCB by their predators. Uptake of PCB from sediment has been reported for shrimp and crabs (Nimmo et al. 1971) and from sea water and sediment by two polychaete worms (Courtney and Langston 1978; Fowler et al. 1978; Elder et al. 1979). In our study, the polychaete worm, Nereis virens, and the shrimp, Crangon septemspinosa were exposed to Aroclor 1254 in sandy sediment (McLeese et al. 1980a). In these experiments, water flows were maintained in the exposure tanks (90% replacement in 1.5 h) to minimize uptake of PCB from the water phase. The concentration of PCB accumulated by the worms was directly related to PCB concentration in the sediment and to exposure time, and was inversely related to worm size. Extrapolation of the data for worms exposed to 0.58 μg PCB/g sediment indicated that final equilibrium concentrations (after 80 d or more) would be about 13.7 μg PCB/g for small worms (CF 24) and 2.3 μg PCB/g for large worms (CF 4). No excretion was detected during 26 d post-exposure.

The shrimp exposed to PCB in sediment gave similar results except that uptake rates were lower and, in the same period, PCB accumulation was about 60% less than that for worms.

The route of uptake for worms could be from direct contact with the sediment or more likely from contact with interstitial water or from PCB adsorbed to ingested sediment. The route for shrimp, with lower uptake rates and less direct contact with the sediment, is more likely to be directly from water or from contact with suspended particulate matter. In other words, the experiments provided indications but did not resolve the question of the major routes for uptake from PCB by these organisms.

LETHALITY OF CHLORINATED HYDROCARBONS IN SEDIMENT AND IN WATER

The lethality of chlorinated hydrocarbons in water, expressed as 24- or 96-h LC50's, for most marine and freshwater organisms reported ranged from about 0.1 μg/L to 1.0 mg/L with a few below and above this range (Klapow and Lewis 1979). Information on lethality of such compounds in sediment seems to be limited to two studies where Aroclor 1254 at high concentrations in sediment did not kill crabs or shrimp (60 mg/kg, Nimmo et al. 1971) or minnows (500 mg/kg, Halter and Johnson, 1977).

Our experiments to compare the lethality of eight chlorinated hydrocarbons in water and sediment to shrimp indicate that PCB dissolved in the water is the more important factor (McLeese et al. 1980, unpubl.). The 96-h LC50's for PCB in water ranged from 0.2 μg/L for endosulfan to 11.6 μg/L for Aroclor 1254. With PCB in sediment, the range was from 4.1 μg/kg for

dieldrin to more than 3400 µg/kg for Aroclor 1254.

In some of the tests with sediment, concentration of chlorinated hydro-carbons in the overlying water was measured. Lethality data (LT50's) from these tests closely fitted the lethality lines for the same compounds in water. This indicates that lethality of chlorinated hydrocarbons present in sediment depends largely on the concentration of the compounds desorbed from the sediment and therefore available in the water column. For comparative purposes, parallel experiments with the worm N. virens are planned.

FIELD COLLECTION OF ANIMALS

Analyses of N. virens collected in the field indicate that there is a difference in PCB, ΣDDT and dieldrin concentration with size, smaller worms having accumulated higher concentrations than large worms (McLeese et al. 1980a). These data confirm size effects noted in N. virens and C. septemspinosa in the laboratory experiments. In terms of bioavailability, the size effect indicates that relative surface area of the animal, metabolic rate or a combination of the two are important.

Field collections of sediments and associated crustaceans, polychaetes and molluscan bivalves from three areas in New Brunswick, Canada, were analysed for PCB, ΣDDT, dieldrin and polyaromatic hydrocarbons (McLeese et al. 1979). For most compounds and species, the concentrations within the animals were relatively constant regardless of the concentrations in the sediments. It was concluded that the concentrations within the organisms were more dependent on the concentrations in the water than in the sediment.

UPTAKE OF CHLORINATED HYDROCARBONS

Marine organisms concentrate chlorinated hydrocarbon compounds to a greater extent from water than from food (Table 1). However, Bryan (1979) concluded that food is the main source of the compounds in fish and crustaceans because the concentrations of such contaminants in sea water are low.

Table 1. Summary of concentration factors of chlorinated hydrocarbons from food (CFF) and water (CFW) for several species.

Compound	Species	CFF or CFW	References
Aroclor 1254	Fish	CFF 1 to 7	Mayer et al. 1977; Freeman et al. 1978
	Fish	CFW 61,000 to 100,000	Mayer et al. 1977; Vieth et al. 1979
	Large Nereis	CFW 50 at 24 h	Calculated from McLeese et al., in press
	Small Nereis	CFW 500 at 24 h	Calculated from McLeese et al., in press.
Endrin	Crab	CFF 5 to 7	Petrocelli and Anderson 1975
	Fish	CFW 1500	Neely et al. 1974
	Oyster	CFW 2700	Mason and Rowe 1976

In our work (McLeese et al. 1980b), lobsters were fed mussels contaminated with a tetrachlorobiphenyl (TPCB), an hexachlorobiphenyl (HPCB), and endrin. Based on measured concentrations of these compounds in the hepatopancreas of the lobsters, estimates of efficiency of uptake at a common level of feeding (9.4 µg of contaminants per feeding) and during the uptake phase were 45, 75 and 71% for TPCB, HPCB and endrin respectively. Estimated concentration factors, were 2, 10 and 1 for TPCB, HPCB and endrin, respectively. In the same order, estimates of time to 50% clearance were 4, 17 and 4 wk. The literature and these results indicate that the potential for accumulation of chlorinated hydrocarbons by aquatic organisms is not as great from food as from water, although, in reality, food may be a more important source.

HEAVY METALS

Marine invertebrates can accumulate heavy metals from sediments. However, the processes controlling the bioavailability of heavy metals from marine sediments are poorly understood.

Biogeochemical factors may affect the bioavailability of the metals to marine organisms. In coastal systems, sediments may become suspended due to turbulence, enhancing metal exchange between sediments, particulate matter and the water, thereby, promoting redistribution of the elements. It is also known that polychaete worms play an important biogeochemical role in the diagenesis of estuarine sediments (Rhodes 1967; Gordon 1966; Young 1968). Sediment texture and composition, salinity, seasonal variations and microbial activity among others may affect the process.

Selected biogeochemical processes affecting bioavailability of heavy metals in marine sediments are discussed below.

CONCENTRATION IN SEDIMENT

Goldberg et al. (1978) have suggested that bivalve molluscs (especially mussels) are suitable indicators for pollution in coastal areas because of their sessile mode of life and their high bioaccumulation capacity for several pollutants. Mussels of the genus Mytilus have been found particulary suitable for this role since they are distributed widely in different temperature regimes of both hemispheres.

Heavy metal contents of benthic animals in some cases have been related to concentrations of the metals in the sediments (Ayling 1974; Ratkowski 1974; Bryan 1974; Bryan and Hummerstone 1978; Bryan and Uysal 1978). Bryan (1974) studied Cu, Zn, Cd, Pb, Ag and As concentrations in N. diversicolor collected from different estuaries where metal concentrations in the sediments varied as much as two orders of magnitude. Cu, Pb, Cd and Ag concentrations in the animal tissues were roughly proportional to those in the sediments. The level of Zn did not change. It was also shown that the rate of Cu accumulation is higher in animals collected from sediments with high Cu levels, suggesting the animals have a higher chelating capacity in the body surface and thus they can store higher amounts of Cu.

In a laboratory study, Ray et al. (1980) found that Cd concentration in

N. virens exposed to Cd-spiked sediment increased linearly with time and with concentration of Cd in the sediment over a 24-d exposure period.

However, others have provided field and laboratory data suggesting that the metal concentrations in the animals do not always reflect the metal concentrations in the sediments from which they were collected. Neff et al. (1978), in an extensive laboratory study, exposed 5 different test organisms to three natural sediments for periods up to 6 wk to determine if the sediment associated metals (Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn by all species and Hg and V by selected species) were bioavailable to the test animals. Out of 136 test combinations, only 36% showed statistically significant relations between the heavy metal concentration in the sediment and in the tissues of the exposed animals. Cross et al. (1970) observed that in six species of polychaete worms collected from sediments containing substantially different quantities of Fe, Mn and Zn, the levels of the elements in the tissues remained essentially constant, indicating that the worms regulate these metals. Luoma (1977) found that concentration of Hg in worms (N. succinea) and the shrimp (P. debilis) exposed to estuarine sediments was very low compared with the concentration in the sediment and the concentration factors (animal/sediment) were only 0.01-0.08 over a period of 21 d. Ray et al. (1979) showed that in shrimp (C. septemspinosa) and bivalve (M. balthica) collected from several locations, the bio-accumulation of Cu, Zn, Cd and Pb in the animals was low under natural circumstances. The highest and lowest metal contents in the sediments were in the ratios of approximately 14, 30, 100 and 100 for Cu, Zn, Cd and Pb, respectively. The corresponding ratios for shrimp were 1.5, 1.1, 1.3 and 5.3 for Cu, Zn, Cd and Pb, respectively. In the same order, the values for Macoma were 2.0, 7.0, 4.5 and 73. These observations indicated that the concentrations of the elements within the animals do not reflect sediment metal concentration except in the case of M. balthica for Pb. In a subsequent study, Ray et al. (in press) exposed three bottom-dwelling invertebrates to two naturally contaminated sediments for 30 d to study bioaccumulation of Cu, Zn, Cd and Pb. M. balthica showed increase in tissue concentration of all four metals from one sediment and only in Cu and Pb from the other. Concentration of Cu and Zn in N. virens and C. septemspinosa exposed to either sediment remained constant. Crangon showed an increase in Pb concentration in animals exposed to both sediments while Cd in the tissues remained constant. N. virens, on the other hand, showed an increase in Cd and Pb when exposed to one sediment but not the other.

The inconsistent relationships observed between metal concentrations in the sediment and the animal tissues do not permit definitive prediction of heavy metal bioavailability on the basis of total metal concentrations in the sediment.

EFFECT OF CHEMICAL FORM OF HEAVY METAL

Ambiguous relationships observed between the metal contents of the animal tisuses and the sediments focused attention on the forms in which metals are associated with the sediments.

One of the problems of adequate assessment of the bioavailability of heavy metals is the difficulty of distinguishing between natural or anthropogenic input. Metals in the crystal lattice of the minerals are not believed to be available to the ecosystem. Adsorption of heavy metals on hydrous iron or manganese oxides or on detrital materials also decreases

their bioavailability (Jenne and Luoma 1977).

Thermodynamic considerations lead to the conclusion that bioavailability should depend on free metal ion activity - the driving force for chemical reactions. Bioaccumulation of heavy metals involves the transport of their ions or complexes to receptor sites in biological membranes, binding to receptor sites and transfer through the membrane. The surface layer of the sediment is likely to play the most significant role in the transport to receptor sites and the concentration of heavy metals in this layer may not be the same as the concentration in the bulk of the sediment. In most cases, only free metal ions are likely to interact with the receptor sites and the extent of binding depends on the free metal ion activity. The interaction may be in competition with other heavy metal ions present and, in turn, will be controlled by relative metal ion activities and relative stability constants of the various metal-ligand combinations.

The bioavailability of free metal ions is also affected by sorption, precipitation, complexation hydrolysis and other geochemical processes occurring simultaneously.

Jenne and Luoma (1977) provided evidence that the bioavailability of trace metals depends upon the equilibrium solute concentration in the associated waters due to sorption-desorption and dissolution-precipitation reactions. The concentration of Cd in N. virens was related to the concentration of the element leached into the water (Ray et al. 1980). The bioavailability of Cu, Zn, Cd and Pb was related to the amounts of metals leached into the water.

Hg, As and possibly Se may be converted to volatile organo-metallic compounds and hydrides due to microbial activity within the sediments and can be released to the water.

Attempts have also been made to distinguish the available metal concentration from the total metal concentration by extracting the sediments with reagents that are neutral, weakly acidic or basic to create redox potential changes, or to provide a chelating ligand for stable complex formation.

Luoma and Jenne (1976) devised procedures for determining the bioavailability of Ag, Cd, Co and Zn sorbed to particles of different chemical composition (iron and manganese oxides, precipitated carbonates, biogenic carbonates and organic detritus) to the deposit-feeding Macoma balthica and related it to binding strength between the metals and the particles. Ag was bioavailable from all sediments but the uptake of Cd, Co and Zn depended upon their physico-chemical form. Extraction of sediment-bound Cd and Co by 70% ethanol or 1 N ammonium acetate provided good estimations of their bioavailability. Extraction with 1 N ammonium acetate or 1 N NaOH plus EDTA provides best estimates for bioavailable Zn. Amount of Cd, Co and Zn extracted from sediments with weak acids (0.1 N HCl; 25% CH_3COOH), reducing agents (1 N hydroxylamine hydrochloride) or oxidizing agents (3% H_2O_2 plus citrate) correlates poorly with bioavailability.

Organo-mercury compounds, containing a covalent metal-carbon bond, are taken up much more rapidly by the animals than the metal chelates containing coordinate linkages. The concentration factor for methyl mercury in the

euphausiid <u>Meganyctiphanes</u> <u>norvegica</u> reached 10^4 after 28 d and was still increasing, while that for inorganic mercury levelled off at a value of 2.5×10^2 (Fowler 1976). The euphausiids also retained the methylated mercury much more. Only 10% was excreted over a period of 3 wk, whereas, 90% of the inorganic mercury was eliminated in the same period. Chipman et al. (1968) showed that the polychaete <u>H. hystrix</u> could accumulate Mn from organic detritus but not from that associated with inorganic matter, suggesting that the form in which the metal is associated with the sediment is the dominating factor.

Luoma and Bryan (1978) found that the concentrations of Pb in the bivalve <u>Scrobicularia plana</u> exposed to sediment bound Pb is controlled by the concentration of Fe and depends on the Pb/Fe ratio in the 1 N HCl extracts of the sediments. Cooke et al. (1979) examined bioavailability of Cd to the cockle, <u>Cerastoderma edule</u>, from four sediments prepared in the laboratory and found that significant uptake of Cd occurred in the tissues of the animals exposed to the biogenic calcium carbonate sediments which, unlike the other three sediments, had an appreciable amount of Cd in the overlying water. Luoma and Bryan (1979) have suggested that bioavailability of sediment-bound Zn to <u>Scrobicularia plana</u> and <u>Macoma balthica</u> depends upon ammonium acetate extractable Zn. Organic materials greater than 1% in the sediment also affected bioavailability.

Selective chemical extraction can distinguish between the different chemical forms in which the heavy metals are bound to the sediment and it seems to be a promising method for assessing bioavailability.

SIZE EFFECT OF ANIMALS

Variation in bioaccumulation due to varying body size has been observed. Ayling (1975) in a study with a single population of <u>Crassostrea gigas</u> noted that smaller oysters contained higher amounts of Cd. Boyden (1977) examined the effect of body size on contents of eight elements in a variety of molluscs. A double logarithmic plot of element content against body weight produced straight-line relationships falling in two categories with slopes of 0.77 and 1.00. In the first case (e.g. Zn in <u>Mytilus edulis</u>) the smaller animals have higher concentrations of elements, while in the second (e.g. Cd in <u>M. edulis</u>) the concentrations were independent of size. <u>Patella vulgata</u> had highest Cd concentration in largest individuals. Ray et al. (1979) showed that small <u>N. virens</u> accumulated higher amounts of Cd from spiked sediment than large ones. The rate of bioaccumulation for the larger worms was also lower in all cases than for the smaller worms and by 24 d, depending upon Cd concentration, the Cd levels in smaller animals were 28-54% higher than in larger worms.

Because of the demonstrated size effects, information regarding bioavailability must be corrected if the samples cover a wide size range of animals.

SEASONAL VARIATION

Seasonal variation in the heavy metal body burden has been observed. Bryan (1973) observed the seasonal variation in scallops, <u>Pecten maximus</u> and <u>Chlamys opercularis</u>, and found that the highest values for seven metals studied occurred in fall and winter. Luoma (1977) noted that in

shrimp, Palaemon debilis, Hg concentration fluctuated over two orders of magnitude during the year. Boyden (1977) pointed out that in Patella, where a single population was sampled, Cu, Pb, and Zn remained fairly constant, but the Cd content increased from 401 to 717 μg Cd/g from March to January. Bioavailability studies can be strictly compared only if such seasonal variations are taken into account.

CHARACTERISTICS OF ORGANISMS

Heavy metal contents of sediment are likely to affect mainly the sediment-ingesting animals such as polychaetes. Filter feeders are affected by the metal contents of the suspended particulate matter. Because of such differences, the rates and extent of bioaccumulation may vary significantly among different species. In addition, the extent of bioaccumulation (Table 2) will depend upon the relative ability of the animals to depurate the metals.

Table 2. Concentration factors for trace element composition of shellfish relative to the amount present in sea water (from Ketchum 1972).

Element	Concentration factors x 10^3		
	Scallop	Oyster	Mussel
Cadmium	2,260.0	318.0	100.0
Chromium	200.0	60.0	320.0
Copper	3.0	13.7	3.0
Silver	2.3	18.7	0.3
Lead	5.3	3.3	4.0
Zinc	28.0	110.3	9.1

Closely related species within the same order can accumulate different amounts of Cd from sediment (Jackim et al. 1977).

BIOAVAILABILITY FROM WATER

Questionable relationships observed for total metal content of sediments as a direct source of heavy metals have suggested water and food as other alternatives. Bioaccumulation can occur through an intermediate stage, for example, from water. Several authors have documented the a priori requirement of metal leaching into the water column for bioaccumulation. Ray et al. (1980) have observed that bioaccumulation of Cd by N. virens from contaminated sediment primarily occurs through Cd leached into the water from the sediments and the amount of Cd uptake was related to the concentration of Cd leached into the water. In a subsequent study (Ray et al., in press) with two contaminated sediments, it was shown that N. virens accumulated Cd from only one sediment, that which produced a trace amount of cadmium in the overlying water. Ray and McLeese (1980) have shown further that the amount of metal leached into the water depended upon the exchange capacity and the organic carbon contents of the sediments. The importance of the release of heavy metals from sediments to the associated water, allowing bioaccumulation of the metals by marine invertebrates, has been further documented (Holmes 1974; Jenne and Luoma 1977; Luoma and Jenne 1977; Cooke et al. 1979; Kniep and Hazen 1979).

SUMMARY

Bioavailability of chlorinated hydrocarbon compounds and heavy metals to marine organisms is not well understood. It appears that the concentration of these compounds in water is the crucial factor determining their bioaccumulation, or toxicity to, benthic fauna.

The main sediment specific process controlling the concentration of chlorinated hydrocarbons and heavy metals in water is the adsorption/desorption equilibrium. Additional factors such as hydrolysis, complexation, chemical reactions, and competition for binding sites control the concentration of heavy metals in water. Site-specific processes such as water currents, microbiological and macrobenthic activity are additional factors influencing the concentration of chlorinated hydrocarbibs and heavy metals in water.

The analysis of "bulk" sediment provides only limited indications of its potential to be a source of chlorinated hydrocarbons and heavy metals to benthic fauna. Bioaccumulation studies and modified elutriate tests are better indicators of bioavailability, but there is a need for simpler and faster techniques. Selective extractions of heavy metals are a promising possibility.

Organochlorine compounds are very strongly bound to the sediment and it appears that sediments are not likely to be a major source of contamination of benthic fauna, except when sediments are very highly contaminated.

The possible contribution of accumulation from direct contact with sediments or fine particulate matter has not been properly assessed or ruled out. The availability of organochlorines and heavy metals from contaminated food, in terms of uptake rates or accumulation, usually is considerably less than their availability from water. However, because of low concentrations in water, food may be a major source of contaminants, particularly for organisms at the higher trophic levels or for pelagic organisms that usually live above the sediments.

The regulated limits of scheduled substances under the Ocean Dumping Control Act of Canada provide sufficient protection of the marine environment, but further research is needed to translate the toxicity-based criteria for chlorinated hydrocarbons into some more easily measurable parameters and to improve our understanding and predictive ability of sediment/fauna interactions.

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