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BIOLOGICAL AVAILABILITY OF SEDIMENT-BOUND TRACE METALS.

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**ABSTRACT.** The impacts of trace contaminants in aquatic environments cannot be assessed realistically without a greater understanding of the factors controlling the biological availability of the concentrated pool of metals associated with sediments. It has been established that direct uptake of (at least some) trace metals from sediments contributes to metal concentrations in benthic organisms. Important controls on metal uptake from sediment are the concentration of the exposure, the partitioning of the metals among components of the sediment, and the redox potential of the sediment. Important problems in ecology, physiology, geochemistry and biogeochemistry remain unresolved, however, and a concentrated interdisciplinary research effort will be necessary before a complete understanding of the fate and impact of sediment-bound metals will be possible.

#### INTRODUCTION

One important objective of studying trace metal biogeochemistry is to develop means of predicting or understanding the biological impact of these contaminants, based upon their concentrations in the environment. This will require understanding both biological and geochemical aspects of trace metal behavior.

When a metal is released in an aquatic system it immediately distributes between particles and solution. Concentrations bound to particles (sediments or suspended particulates) are usually orders of magnitude higher than concentrations in solution. This paper is a summary of some of the work we have done studying the processes that determine the extent of transfer to food webs of metals within this concentrated particulate-associated metal reservoir.

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## 1. SEDIMENT GEOCHEMISTRY OF METALS

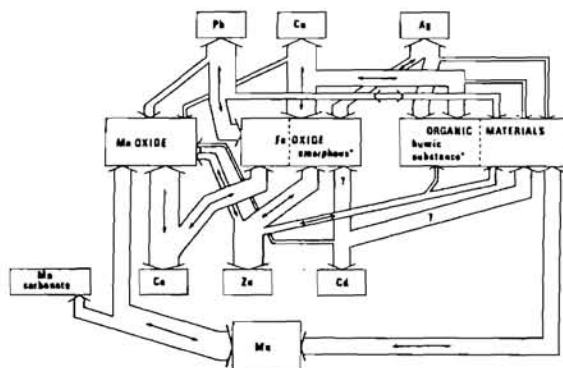
Particles in estuarine environments are composed of a number of components capable of strongly binding trace metals. In oxidized sediments (which will be considered exclusively in this paper) these include oxides of iron, oxides of manganese, various forms of organic metals and clays. Most particles are aggregates of all these components (Jenne 1977). Most of the organic materials, iron oxides and manganese oxides occur as coatings often covering clay surfaces. By themselves, clays also bind metals less strongly than the other components (Davies-Colley et al 1985). The combination of a lower binding intensity and a loss of binding sites to interactions with other components suggests clays are much less important than other types of components in binding metals in sediments (Jenne, 1977, Davis-Colley et al., 1984).

The distribution of a metal among the components of the sediments is determined by three factors: (1) The intensity of metal binding to each component; (2) the number of available binding sites per unit mass of each component; and (3) the mass of each component per unit mass of sediment. By quantitatively characterizing each of the above a mathematical description of metal distribution within a given sediment should be possible (Oakley et al., 1982; Luoma and Davis, 1983; Davies-Colley 1984; Tessier et al., 1984). However, a number of the prerequisites for an accurate model are poorly known. Poorly understood factors include (1) measures of binding intensity that are comparable among components; (2) the effect of component interactions within aggregates upon the number of binding sites; (3) the constancy of binding intensity at different metal concentrations per unit component; and (4) how to convert measures of component concentrations to number of component binding sites per unit mass of sediment (Luoma and Davis 1983). Thus, model calculations of metal partitioning (Davis-Colley, et al, 1984; Tessier, et al., 1984, Luoma, in press) must be considered simplistic approximations.

In lieu of a technique for precisely modeling metal partitioning in sediments, operational techniques have been developed. A large number of leaching techniques have been applied to aquatic sediments (Gibbs, 1973; Forestner 1979; Tessier, 1979). Nearly all suffer from a lack of chemical specificity (Luoma and Jenne, 1976; Guy, et al., 1978); and few, if any, efficiently extract metal from a single component of the sediment.

Statistical techniques also are employed to assess partitioning (Luoma and Bryan 1981) but these are qualitative and subject to the errors of any correlative approach, especially those associated with confounding variables.

Despite their inadequacies, some consistent conclusions emerge from the different approaches to studying metal partitioning. The clearest is that most metals distribute among several components or types of binding sites in oxidized sediments. It also appears that this distribution can change if the chemical characteristics of the sediments change. Leaching studies consistently show that several metal fractions of different extractability occur in almost all sediments. A



\*Operationally-defined

Figure 1. Partitioning of Ag, Cd, Co, Cu, Pb and Zn among binding components important in oxidized sediments, as indicated by statistical associations (from Luoma and Bryan, 1981).

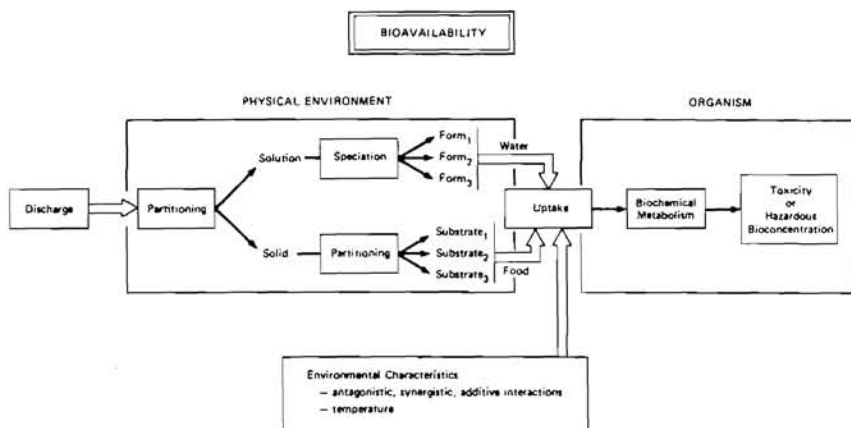


Figure 2. A simplified schematic illustration of the various processes which affect the availability of trace contaminants to organisms.

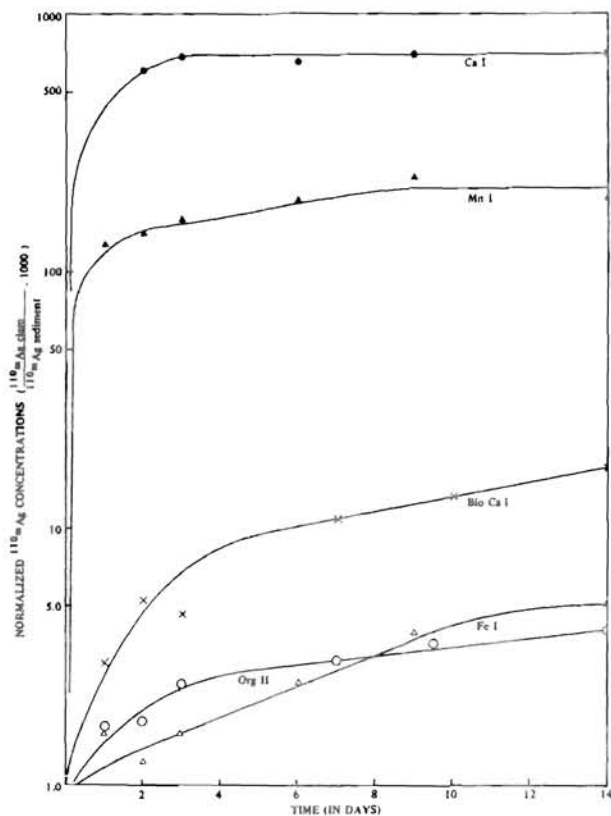


Figure 3. Differences in uptake of  $^{110m}\text{Ag}$  when different types of well-defined sediments were ingested by the deposit-feeding clam *Macoma baltica*. Sediment-types are amorphous iron oxides (Fe), decaying marsh grass fragments (org), manganese oxides (Mn), crushed bivalve shells (BioCa), and calcium carbonate (Ca) (from Luoma and Jenne, 1977).

statistical study, assessing correlations of metal concentrations with component concentrations among nearly 50 geochemically diverse sediments strongly indicated the multi-component nature of metal binding (Fig. 1; Luoma and Bryan). More recently, attempts to calculate theoretical metal distributions have shown that at least Cu is mainly distributed between iron oxide and organic materials in sediments, but the relative importance of each differs among sediments (Davies-Colley 1985; Luoma, in press). This means that no single component dominates the partitioning of a metal; and that partitioning may change from place to place or time to time.

## 2. BIOLOGICAL IMPACT OF VARIABLE METAL PARTITIONING.

If metals partition among a number of components, then organisms that ingest or contact sediments are not exposed to the sediment-bound metal as a single entity. Rather, each metal is encountered in a variety of metal-ligand associations (Fig. 2); and that variety is not constant in time or space. Laboratory studies clearly show that each metal-component association has a distinctly different biological availability. Cadmium is accumulated rapidly by deposit feeding organisms (the clam Macoma balthica) from sediments containing organic materials such as the polysaccharide exopolymer secreted extracellularly by some bacteria (Harvey and Luoma, 1985). However, Cd is not accumulated from sediments stripped of organic materials or from particulates that are either purely iron oxides or are composed of iron oxide coated with living bacteria (Luoma and Jenne 1976; Harvey and Luoma, 1985). The rate of uptake of Zn, Ag and Co may differ by orders of magnitude when these metals are bound to different specific components of the sediment (Luoma and Jenne, 1977). For example, Fig. 3 shows that uptake of Ag by M. balthica was 10 fold greater when manganese oxide-bound Ag was ingested as compared to ingestion of Ag associated with iron oxides or organic materials (marsh grass detritus).

Thus, geochemical studies show that the distribution of a metal among components may vary from sediment to sediment, and laboratory biological studies show that a difference in component association of a metal may cause a difference in metal bioavailability. Therefore, in nature metal concentrations in organisms should vary among sediments of differing component concentrations.

## 3. COMPARISONS OF METALS IN ORGANISMS AND SEDIMENTS.

In natural systems organisms are directly exposed to sediment-bound metals through surface contact, and accidental or intentional ingestion during feeding. Sediments also may control solute metal concentrations in waters in contact with the sediment bed sufficiently long to approach equilibrium, thus affecting biological exposures through the solute vector of uptake. Thus, metal tissue burdens in deposit and suspension feeding organisms should directly reflect the biological availability of sediment-bound metals. These organisms are exposed to all components of the sediment in their quest for nutrition. Most choose their food only on the basis of particle size, or particle density (Newell, 1965, Whitlatch, 1974; Bubnova, 1974, Self and Jumars, 1978), although particle-size selectivity may differ among species. For example the clam, Macoma balthica,

digests various types of particles less than 80  $\mu\text{m}$  in size (Yonge, 1949). The average particle size in the digestive gland of *Macoma inguinata* is 10  $\mu\text{m}$ , while particles in the gut of *Macoma secta* average 300  $\mu\text{m}$  (Reid and Reid, 1969). The more selective feeder, *Pectinaria gouldii* (a polychaete worm), ingests particles <75  $\mu\text{m}$ , and >25  $\mu\text{m}$ , but employs no detectable chemical selection (Whitlatch, 1974). Selective suspension-feeding organisms also may inadvertently ingest a variety of metal-binding substrates when heavy concentrations of re-suspended materials are mixed with phytoplankton in the water column, or may employ some suspended sediment as an essential source of nutrition.

Most benthic organisms also are exposed primarily to metals associated with oxidized particles (Luoma and Davis, 1983). In oxygenated waters, reducing conditions occur in sediments below the sediment-water interface, but a thin layer of oxidized sediment is left at the sediment-water interface. Many infaunal organisms feed at the oxidized sediment-water interface, or irrigate borrows with water from the oxidized surface. Meiofauna live exclusively above the redox interface (Coull, 1979), and epibenthic organisms are in contact, almost exclusively, with the oxidized environment at the surface of the sediment. Where reducing conditions occur, they undoubtedly affect the availability of sediment-bound metals; but in general the oxidized surface layer of sediments is much more important biologically than indicated by its mass.

If exposures to sediments control bioaccumulation by benthos, then metal concentrations in organisms in nature might correlate with concentrations in sediments. Strong correlations are most commonly not observed, however. Poor correlations between marine or estuarine benthic organisms and their sedimentary habitat have been reported by Cross et al., (1970), Halcrow, et al., (1973), Valiela et al., (1974), Huggett et al., (1975), Luoma (1977b), and Schnell and Nevessi (1977). The poor correlations between metal concentrations in benthic organisms and sediments could mean that metal in benthos are not derived from sediments, or sources coupled to sediments. However, they could also result from statistical problems in the comparison or from the influence of variables other than sedimentary metal concentrations upon metal bioavailability.

Two especially important problems have characterized statistical comparisons of metal concentrations in benthos and their food: (1) the actual food of the organisms has not always been used in the comparison; and (2) an adequate data range has not always been employed.

Obviously, metal concentrations in filter-feeding organisms such as oysters, mussels and some species of clams (that are dependent primarily upon phytoplankton for food) or in herbivorous grazing animals, would not necessarily correlate with metal concentrations in sediments, even if food were the primary vector of metal uptake. For example, concentrations of most metals in grazing snails follow concentrations in algae, but not sediments, where sediments and algae are not correlated (Bryan and Hummerstone, 1977; Young, 1975). Even among deposit-feeding

organisms, differences in food sources may affect metal exposure. For example, Phelps (1967) and Phelps, et al., (1969) reported consistent differences in Zn concentrations among benthic polychaetes from different feeding guilds (surface feeders vs. subsurface feeders; selective vs. non-selective feeders).

Most correlation studies also have been conducted within a single body of water, including only narrow concentration gradients in the comparison. Comparisons which have considered wider data ranges have shown some significant correlations between metal concentrations in deposit feeders and concentrations in sediments. Bryan (1974) found that concentrations of Cu, Pb, and Cd in the polychaete N. diversicolor correlated strongly with concentrations in sediments from the sediment-water interface when data were collected from several estuaries. Packer et al (1980) found that Cd and Zn concentrations in the polychaete Arenicola marina followed Cd and Zn in sediments from 24 stations on the coast of Wales. Significant correlations were not observed for Pb, Cu, and Mn, however. In an extensive survey, Luoma and Bryan (1978, 1979, 1982) compared concentrations of Ag, Cd, Co, Cu, Pb and Zn in two deposit feeders, the burrowing clam Scrobicularia plana and the polychaete N. diversicolor, to concentrations in sediments over a wide range of conditions among 50 stations in 17 estuaries. The data range for all metals was two to three orders of magnitude. Statistically significant (but weak) correlations between total metals in sediments and the tissues of S. plana were observed for Pb (Fig. 4), and for all other metals except Cu. Stronger

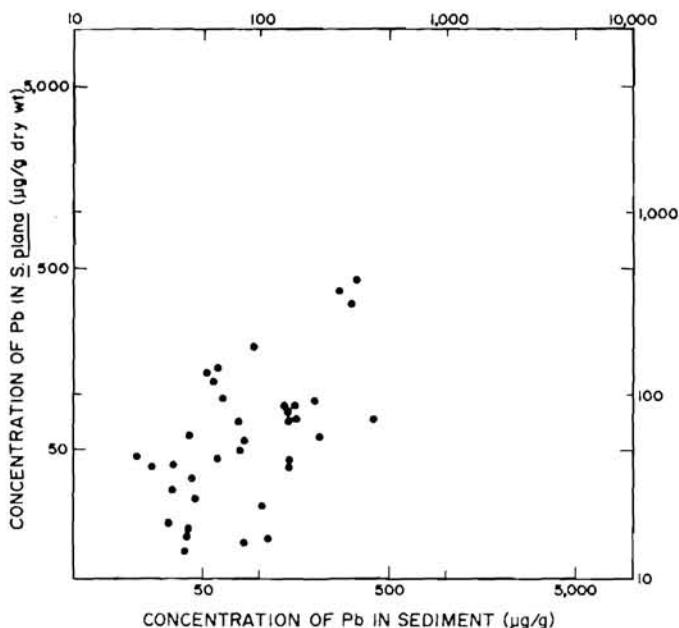


Figure 4. Correlation of Pb in surficial estuarine sediments with Pb in the deposit-feeding bivalve Scrobicularia plana.  $r = 0.69$  for power function fit. Data from 17 estuaries (from Luoma and Bryan, 1978).

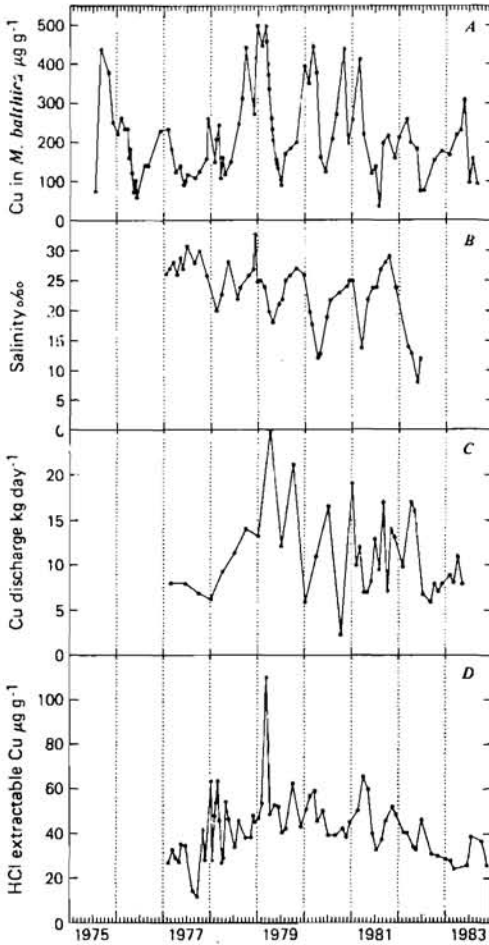


Figure 6.

Correlation of Pb in *S. plana* with ratio of Pb/Fe (both extracted by 1N HCl) in surficial estuarine sediments.  $r = 0.95$ . Data from 17 estuaries (from Luoma and Bryan, 1978).

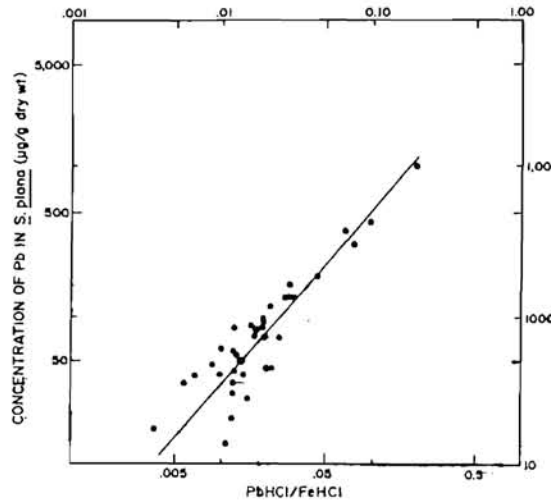


Figure 5.

- A) Concentrations of Cu observed in the soft tissues of *M. balthica* in South San Francisco Bay between June 1975 and June 1983. Representative errors bars (one SEM) are shown in 1979.
- B) Salinity observed at same station in water in the mantle cavity of *M. balthica*.
- C) Cu discharge from the sewage treatment works located 1 km south of above station.
- D) HCl-extractable Cu in surficial sediments (<100µm grain size) at same station.



correlations were observed between sediments and N. diversicolor for Cu and Pb. However, over the broader data set, these correlations were not as strong as observed by Bryan (1974.) In general, the data suggested that total concentrations contributed to metal concentrations in the deposit feeders, but that an understanding of other contributing variables would be necessary to predict metal concentrations in organisms from concentrations in the environment.

Sediments need not be the only source of metal exposure for benthic organisms. Deviations in correlations between organisms and sediments could result from uptake from solution. However, even where the source of exposure which controls both the food and solute vectors is well-defined, deviations of bioaccumulation from dependence upon exposure alone are evident. For example, Bryan and Hummerstone (1977) observed differences in Ag and Cu concentrations in S. plana from two estuaries that could not be explained by Ag and Cu either in water or in sediment. In San Francisco Bay, spatial variations in Cu concentration indicated a sewage outfall was the primary source of the metal for the burrowing clam M. balthica (Thomson et al., 1984). However comparisons of nine years of near-monthly analyses of plant effluent, and clams from this site showed that, although Cu levels in M. balthica were occasionally coupled to Cu discharges, large fluctuations in concentrations occurred in the clam which could not be explained by changes in exposure (Fig. 5). Again, a general dependence upon exposure was evident, but other variables were also obviously important in controlling bioaccumulation.

Recent studies strongly indicate that one of the most important of those factors is the chemical characteristics of the sediment. The importance of such characteristics was first clearly demonstrated by Luoma and Bryan (1978). As previously mentioned, concentrations of Pb in the deposit feeding bivalve Scrobicularia plana were significantly correlated with concentrations of Pb in sediments from 37 stations in 17 estuaries in southwest England (Fig. 4) in that study. However, the correlation was characterized by substantial variability. It was noticed that the stations with unusually high Pb concentrations were stations where the ratio between Pb and extractable Fe\* was also high. Among all stations concentrations of Pb in S. plana were very closely correlated ( $R^2 = 0.89$ ) with Pb/Fe in sediments (where Fe was measured by HCl extraction, Fig. 6).

Luoma and Bryan (1978) suggested that the availability of Pb was enhanced at low concentrations of Fe in sediment because less Pb was strongly bound to iron oxides as the Pb/Fe ratio in sediments increased making more Pb biologically available in the digestive tract. Whatever the cause, the ratio Pb/Fe, in sediments was an accurate predictor of Pb in S. plana in estuaries not included in the original regression calculation

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\*Fe extracted either with 1NHCl for two hours or 0.4N acid ammonium oxalate for two hours--presumably an amorphous fraction of iron oxide.

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Estuary	HCl-soluble Pb (ug/g)	Pb observed in <u>S. plana</u> (ug/g)	Pb predicted in <u>S. plana</u> from Pb/Fe in sediment
Axe	25	8	9
Thames	57	25	36
Elorn	71	31	34
E. Looe	93	104	128
Gannel	417	327	330
Humber	191	22	28

Table 1. The concentration of Pb observed in sediments (HCl extraction) and soft tissues of Scrobicularia plana from six estuaries compared with the concentration of Pb predicted for S. plana from the Pb/Fe ratio in the sediments. Data from Luoma and Bryan (1978).

	Best Extraction Method	Vector	Inhibits Availability	Enhances Availability
Ag	HCl	sediment (interstitial water)	Cu extractable Fe?	sewage factor
As	Total	sediment solute (polychaetes)	extractable Fe	
Cd	HCl	solute sediment	anoxia (S=)	sewage factor?
Co	HCl	sediment (solute?)	organic carbon	
Cu	Total HCl	sediment (polychaetes)	extractable Fe organic carbon?	anoxia (molluscs)
Hg		sediment	organic carbon extractable Fe	moderate Eh low organic carbon
Pb	Total HCl	sediment	extractable Fe organic carbon	
Zn	1N ammonium acetate HCl	sediment solute	organic carbon	

Table 2. Summary of various factors which appear to inhibit or enhance the availability of sediment-bound trace metals to benthic organisms.

(Table 1). In estuaries such as the Looe, Pb in S. plana was greater than in estuaries such as L'Elorn in France, although Pb concentrations in sediment were similar, because extractable Fe was lower in the Looe. The most dramatic example was the Humber, which includes the industrial Midlands of England in its watershed. Although sediments were highly contaminated with Pb, the statistical model predicted little Pb should occur in S. plana in the Humber because of the very high Fe concentrations in the sediment. Observations proved this to be the case.

Similar to Pb, Langston (1980; 1982) has shown that levels of As in S. plana and M. balthica are also best related to the As:Fe ratio in a 1N HCl extract of surface sediment. Mercury, on the other hand, has a high affinity for organic particles and concentrations in S. plana and M. balthica are related to the total (HNO<sub>3</sub> extractable) Hg:total organic matter ratios in the <100 um fraction of surface sediments (Langston, 1982). This agrees with the work of Breteler et al. (1981) which showed that the highest concentration of Hg in mussels (Modiolus demissus) and crabs (Uca pugnax) occurred in animals exposed to sediments having low levels of organic matter.

Physiochemical conditions other than sediment component concentrations also appear to influence metal bioavailability from sediment. For example, the availability of Ag (which appears in the sediments of the Seine in concentrations higher than reported anywhere in the world) to deposit feeding bivalves is strongly reduced when high concentrations of Cu appear in sediments (Luoma and Bryan, 1982). Increased pH appears to increase availability of Hg (perhaps by stimulating conversion of Hg<sup>+2</sup> to Hg<sup>0</sup>), but not Cu and Pb to marsh plants (Gambrell et al., 1977). Unpublished data from the study of Luoma and Bryan also suggested Cd was of lower availability in highly anoxic mudflats (e.g., inner Poole Harbor). The availability of Cu to the polychaete Neanthes arenaeodentata also is less from subsurface than from surface sediments (Pesch, 1979), presumably due to differences in redox potential. In contrast, certain types of reducing conditions enhanced the bioavailability of Cu to the clams S. plana and M. balthica (Luoma and Bryan, 1982). Exceptionally high concentrations of Cu were observed in these animals in seven anoxic mudflats, and during an unusual period of anoxia on one mudflat. Greater enhancements of Cu availability generally accompanied lower concentrations of Fe in sediments, but the precise cause of the enhancement was not identified.

In a recent review, Bryan (1985) summarized, for a number of trace metals, the results of his extensive experience, and that of others, with processes controlling metal availability from sediments to aquatic organisms. It must be emphasized that these conclusions are preliminary. Results sometimes differed among species, and definitive studies have only been conducted with a few species, mostly in estuarine environments. From Bryan's summarization and our experience, the following conclusions appear: (1) High concentrations of organic matter and/or Fe (probably as amorphous iron oxide) inhibit the availability of Hg, Pb, As, Zn, and possibly Ag and Cu. Sediments high in total organic carbon or extractable Fe may contain high concentrations of trace metals, but the metals do not appear to pass on to most biota. (2) Under some circumstances or for some species, the

solute vector of uptake is an especially important source of Cd, Zn, and possibly As and Ag. (3) Total concentrations in sediments control Cu availability to polychaetes, but the controls on Cu availability to molluscs are not clear. Anoxia may enhance Cu availability to molluscs, under some circumstances. (4) Zn availability is controlled by readily exchangeable Zn in sediment, uptake from the solute vector, and a negative influence of organic carbon. (5) Most metals (especially Hg, Pb, Zn, As, Cu, and Ag) would be expected to be of high bioavailability in coarser grained sediments enriched by anthropogenic metal input and in wastes (such as smelter wastes or mine wastes) low in organic material or Fe. Some unknown attribute of sewage sludge enhances the availability of Ag and possibly Cd. Table 2 summarizes the conclusions presented to date on factors controlling metal availability from sediments to benthos.

#### 4. RECOMMENDATIONS FOR IMPROVING BIOLOGICAL RELEVANCE OF SEDIMENT TREATMENTS.

It must be re-emphasized that our knowledge of metal bioavailability from sediments is, at best, fragmentary. Definitive studies are less than five years old, have been conducted by only a few research groups, consider only a few species, and nearly all have been conducted in estuaries. Nearly all conclusions rest upon statistical evidence which has not yet been mechanistically clarified by definitive laboratory studies. Additional comparative studies among estuaries, rivers or coastal environments are needed, as are controlled studies, testing some of the hypotheses generated by the statistical work.

Within the limitations of our present knowledge, it is possible to recommend a few practical procedures which could enhance the biological relevance of sediment treatment. However, it must be accepted that these recommendations are preliminary and could change as our knowledge grows. Furthermore, the objective is to recommend the simplest possible procedures, so as to maximize their utility in an operational program.

The simplest, biologically relevant measurements of sediments could include:

- (1) Measurement of total concentrations (or near-total using an  $\text{HNO}_3$ -reflux) of trace metals.
- (2) A simple extraction of sediments with a 0.5N HCl. and analysis of trace metals in the extract.
- (3) Measurement of major sediment components which affect bioavailability.

These include:

- (a) Total organic carbon.
- (b) Concentrations of Fe extracted by HCl. (Acid ammonium oxalate is a more widely accepted alternative for estimating amorphous Fe oxide. However, HCl and oxalate extractions of estuarine sediments usually yield similar results. For the sake of simplicity, measurement of Fe in the same HCl extract used for metals would not result in much loss of information.)
- (c) Total Mn.

Collection of such samples should be from microenvironments of most relevance to organisms of interest in the systems involved. In estuaries such as the Seine, this usually means sediments should be collected carefully from the thin oxidized layer at the sediment-water interface. Sediments should be sieved prior to storage. We employ a 100 um sieve, others suggest 60 um. Sediments also should be frozen as soon as possible if they are to be stored prior to extractions, so as to prevent redox changes which could affect extraction results.

This simple program would provide information about the major constituents that appear to affect metal bioavailability, and concentrations of metals that may competitively inhibit availability. From such a program, general assessments of the vulnerability of different sedimentary environments to accumulation of biologically significant concentrations of trace metals could begin to be possible.

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