

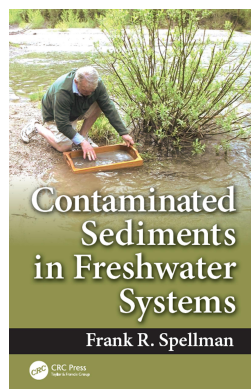
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Sediment Physicochemical Characteristics

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14 Sediment Physicochemical Characteristics

INTRODUCTION

After an environmental practitioner devises a sediment collection plan, determines the sediment collection location, collects sediments, properly packages the collected samples, and labels the sample package contents, the samples are carefully transported to an examination and testing laboratory. Such a laboratory is typically tasked with determining certain physicochemical characteristics of the sediments, in conjunction with toxicity testing or chemical analysis or inorganic or organic contaminants. This characterization should include measurement of certain parameters known to mediate the availability of contaminants in sediment (ASTM, 2000f). Bulk chemical concentrations alone should not be used to evaluate bioavailability (USEPA, 1998). The following parameters are generally measured:

- pH (porewater)
- Ammonia (porewater)
- Total organic carbon
- Particle size distribution (e.g., percent sand, silt, and clay)
- Percent water content
- Salinity and hardness of porewater
- Conductivity of porewater

Depending on the experimental design or study objectives, more extensive characterization may be necessary. Several additional characteristics that may assist in study implementation, data interpretation, or quality assurance/quality control (QA/QC) (e.g., assessing sediment integrity, artifact production, optimal extraction, test procedures) include the following:

- Sediment biochemical oxygen demand (BOD)
- Sediment chemical oxygen demand (COD)
- Sediment oxygen demand (SOD)
- Cation exchange capacity (CEC)
- Redox (Eh) or oxidation-reduction potential (ORP)
- Total inorganic carbon

- Total volatile solids
- Acid-volatile sulfides (AVSs)
- Simultaneously extracted metals (SEMs)
- Metals
- Synthetic organic compounds (pesticides, PCBs, PAHs, TCDD-dioxin)
- Oil and grease
- Petroleum hydrocarbons
- Dissolved organic carbon (DOC) in the porewater

Note that many of the measurements of many sediment physicochemical characteristics use analytical techniques originally developed for soils and waters, and the literature should be consulted for details regarding the recommended methodology (Guy, 1969; Page et al., 1982; Plumb, 1981). The following sections provide the rationale for making each type of sediment physicochemical measurement, as well as a brief description of the measurement techniques for specific procedures.

SEDIMENT PHYSICOCHEMICAL PARAMETERS AND MEASUREMENT

pH

Sediment pH is often one of the single most important factors controlling speciation and equilibria for many chemicals, including sulfides, ammonia, cyanide, and metals, all of which ionize under the influence of pH. The U.S. Environmental Protection Agency (USEPA) ammonia water-quality criterion, for example, is dependent in part on pH because ammonia toxicity is largely governed by the unionized ammonia fraction, which is pH dependent (USEPA, 1999). Metal (Cd, Cu, Ni, Pb, and Zn) speciation and bioavailability are also known to be affected by pH (Ho et al., 1999; Schubauer-Berigan and Ankley et al., 1991).

Generally, pH is measured using a pH meter consisting of a potentiometer, a glass electrode, a reference electrode, and a temperature compensating device. A circuit is completed through the potentiometer when the electrodes are submersed. General-purpose process pH electrodes are available in a wide variety of configurations for inline and submersion applications. Generally, electrodes with gel-filled references require less maintenance than electrodes with liquid-filled references. The latest instruments have microprocessors that automatically calculate and display the slope. Some older instruments have a percent-slope readout or millivolt readout. For instruments with a millivolt readout, the measured electrode potential is calculated as the difference between millivolts measured at the known pH of two buffers.

Plumb (1981) and Gonzalez (1996) described a method for measuring pH in sediment using a pH probe and meter. The probe was inserted into the sediment and pH directly measured after at least a 5-minute equilibration time. Electrodes have also been used for direct measurements of pH in sediment porewater or in a 1:1 mixture of sediment to water (Jackson, 1958). Direct measurement of sediment pH is also possible using electrodes with spear-up designs allowing for greater penetration into the sample (USEPA, 2001).

AMMONIA IN POREWATER

Nitrogen, a nutrient associated with over-enrichment of aquatic environments, exists in several forms, including ammonia. Ammonia is highly soluble in water, where it is found in an un-ionized form as NH_3 and in an ionized form as NH_4^+ . The extent of ionization is dependent on pH, temperature, and salinity (in seawater). Ammonia in sediments and porewater is generally the result of microbial degradation of nitrogenous organic material such as amino acids (Ankley et al., 1990). Porewater concentrations of ammonia as high as 50 mg/L have been measured in otherwise uncontaminated sediments (Kristensen and Blackburn, 1987; Murray et al., 1978), while ammonia in porewaters from contaminated sediments can range from 50 to more than 200 mg/L (Ankley et al., 1990; Schubauer-Berigan and Ankley, 1991).

The toxic effects of ammonia are generally considered to be associated with the un-ionized fraction (NH_3) rather than the ionic components (HN_4^+ and NH_4SO_4^-), which coexist in equilibria. This equilibrium is highly dependent on pH, temperature, pressure, salinity, and ionic concentrations of ammonia. The toxic un-ionized ammonia fraction can be calculated using known total ammonia values and measurements of pH, pressure, salinity, and temperature (Thurston et al., 1981; Whitfield, 1978).

Methods available to measure ammonia in the porewater include the following (APHA, 1995; USEPA, 1983):

1. Titrimetric method
2. Ammonia-selective electrode method
3. Ammonia-selective electrode method using known addition
4. Phenate method
5. Automated phenate method

A preliminary distillation step may be required if interferences (e.g., sample constituents that interact with procedural reagents) are present (APHA, 1995; ASTM, 2000g). Once distilled, the sample can be analyzed using any of the methods listed above.

The distillation and titration methods are frequently used when ammonia concentrations are greater than 5.0 mg/L. The ammonia-selective electrode method is appropriate when concentrations range between 0.03 and 1400 mg $\text{NH}_3\text{-N/L}$. Ammonia readings are calibrated against ammonia standards. To verify meter readings, confirmatory subsamples can be preserved and analyzed for ammonia using the standard Nessler technique described in APHA (1995). For the phenate method, distillation with sulfuric acid is recommended when interferences are present (APHA, 1995). The automated phenate method is suitable for porewaters with ammonia concentrations in the range of 0.02 and 2.0 mg $\text{NH}_3\text{-N/L}$.

TOTAL ORGANIC CARBON CONTENT

The total organic carbon (TOC) content of sediment is a measure of the total amount of oxidizable organic material. TOC is the sum of dissolved organic carbon (DOC), particulate organic carbon (POC) or suspended organic carbon (SOC), and colloids.

TOC is an important parameter to measure in sediments because it is a major determinant of non-ionic organic chemical bioavailability (DiToro et al., 1991). Metal bioavailability is also affected by the amount of TOC present in sediments. TOC is usually expressed as a percentage of the bulk sediment and is used to normalize the dry-weight sediment concentration of a chemical to the organic carbon content of the sediment. The USEPA equilibrium partitioning sediment guidelines estimate bioavailability as a function of contaminant concentration sorbed to sediment organic carbon and contaminant concentration in the porewater under equilibrium conditions (USEPA, 1998). Recently, the presence of soot carbon from the combustion of organic carbon (e.g., fossil fuels) has been recognized as a fraction of the TOC in sediment. Soot carbon may alter the geochemistry and bioavailability of some organic contaminants (Gustuffson et al., 1997).

The organic carbon content of sediments has been measured using several methods, including wet oxidation titration, modified titration, and combustion after removal of carbonate by the addition of HCL and subsequent drying. USEPA methods (USEPA, 1986b, 1987), including SW-846 and 430/0-86-004, are often used to measure TOC. One of two methods may be used to separate organic from inorganic carbon before analyzing for TOC (Plumb, 1981): (1) ignition and using HCl as the acid for pretreating sediment, or (2) differential combustion, which uses thermal combustion to separate the two forms of carbon. Total organic carbon analyses should be based on high-temperature combustion rather than on chemical oxidation (USEPA/USACE, 1998), because some classes of organic compounds are not fully degraded by combined chemical and ultraviolet oxidation techniques. Inorganic carbon (e.g., carbonates and bicarbonates) can be a significant proportion of the total carbon in some sediments. Therefore, samples could be treated with acid to remove the inorganic carbon prior to TOC analysis. The procedure described by the Puget Sound Estuary Program (1987a) is recommended for TOC analysis because this method uses high-temperature combustion using an induction furnace. The USEPA recommends a similar method using catalytic combustion and non-dispersive infrared detection (Leonard, 1991) for quantifying TOC.

PARTICLE SIZE DISTRIBUTION (PERCENT SAND, SILT, AND CLAY)

Particle size is used to characterize the physical characteristics of sediments. Because particle size influences both chemical and biological characteristics, it can be used to normalize chemical concentrations and account for some of the variability found in biological assemblages (USEPA, 1998) or in laboratory toxicity testing (Hoss et al., 1999; USEPA, 2000). Particle size can be characterized in varying detail. The broadest divisions that generally are considered useful for characterizing particle size distributions are percentages of gravel, sand, silt, and clay; however, each of these size fractions can be subdivided further so that additional characteristics of the size distribution are determined (Puget Sound Estuary Program, 1996). Particle size determinations can either include or exclude organic material. If organic material is removed prior to analysis, the *true* (i.e., primarily inorganic) particle size distribution is determined. If organic material is included in the analysis, the *apparent* (i.e., organic plus inorganic) particle size distribution is determined. Because true

and apparent distributions may differ, detailed comparisons between samples analyzed by these different methods are questionable. Therefore, if comparisons among samples between studies is desired, sediment particle size should be measured using constituent methods (Puget Sound Estuary Program, 1996).

Sediment particle size can be measured by a number of different methods (Allen, 1975; ASTM, 2000a; Plumb, 1981; Puget Sound Estuary Program, 1996). The best method will depend on the particle properties of the sample (Singer et al., 1988). Particle size distribution is often determined by wet sieving the sample (Plumb, 1981; Puget Sound Estuary Program, 1996; Singer et al., 1988; USEPA, 1979), the hydrometer method (Day, 1965; Patrick, 1958), the pipet method (Guy, 1969; Rukavina and Duncan, 1970), settling techniques (Sanford and Swift, 1971), or x-ray absorption (Duncan and Lattaie, 1979; Rukavina and Duncan, 1970). The pipet method may be superior to the hydrometer method (Sternberg and Creager, 1961). Combinations of multiple methods may provide refined measurements of particle size distribution. Gee and Bauder (1986) used sieving and pipetting after soluble salts were removed. Gonzalez (1996) used a combination of sieve and hydrometer methods.

Recommended methods for measuring sediment particle size distribution are those of the Puget Sound Estuary Program (1996) and the USEPA (1995). Percent gravel, sand, silt, and clay are determined as apparent distribution using a minimum sediment sample size of 100 g taken from a homogenized sediment sample. Organic matter should be removed prior to analysis by oxidation using hydrogen peroxide. Wet sieving followed by dry sieving (mechanical shaking) separates the two coarse particle size groups. The silt–clay fraction is subdivided using a pipet technique that depends on the differential settling rates of the two different particle size fractions. All fractions are dried to a constant weight. Cooled samples are stored in a desiccator and weighed.

To obtain an accurate determination of particle sizes for the fine fraction, the Coulter (particle size) counter method may be employed (McCabe and Jarvis, 1973; Vanderploeg, 1981). This method gives the fraction of particles with an apparent spherical diameter. In a review of the available methods, Swift et al. (1972) found the Coulter counter method to be the most versatile method overall; however, it does not provide settling information. Another potential method for determining the particle size distribution of a very fine fraction is through the use of electron microscopy (Leppard et al., 1988). Collection techniques for very fine material can result in aggregation of larger colloidal structures (Leppard, 1986; Leppard et al., 1988). In general, particle settling methods are preferred to sediment sizing methods.

PERCENT WATER OR MOISTURE CONTENT

Water content is a measurement of sediment moisture usually expressed as a percentage of the whole sediment weight. It is known to influence toxicity and is used to aid in the interpretation of sediment quality investigations. Sediment moisture content is measured as the difference between wet weight of the sediment and dry weight following oven drying at 50 to 105°C to a constant weight. Percent water is used to convert sediment concentrations of substances from wet weight to a dry weight. Methods for determining moisture content were described by Plumb (1981) and Vecchi (1999). Additional methods are provided in USEPA (1987).

SALINITY OF THE POREWATER (MARINE SEDIMENTS)

Salinity is a measure of the mass of dissolved salt in a given mass of solution. The most reliable method to determine the true or absolute salinity is by complete chemical analysis; however, this is time consuming and costly. Therefore, indirect methods are more suitable. Indirect methods include conductivity, density, sound speed, or refractive index (APHA, 1995). Salinity is then calculated from the empirical relationship between salinity and the indirect measurement. Conductivity measurements have the greatest precision but respond only to ionic solutes (APHA, 1995). Density measurements respond to all solutes. APHA (1995) recommended the electrical conductivity method, because it is sensitive and easily performed, and the density method using a vibrating flow densitometer. USEPA (1986b) methods should also be consulted. A salinity refractometer can be used for quick readings of salt density in solutions such as seawater. These refractometers are easy to read, noncorrosive, and lightweight. They have dual scales and an adjustable focus. Temperature and non-temperature compensating refractometers are available. Most refractometers are accurate to 1 ppt and read specific gravity (1.000 to 1.070 in 0.001 divisions) and parts per thousand (0 to 100 in 1 ppt divisions).

CONDUCTIVITY OF THE POREWATER (FRESHWATER SEDIMENTS)

Conductivity is a measure of the ability of an aqueous solution to carry an electric current. This ability is dependent on the presence of ions in the solution, the concentration of the ions, their mobility and valance, and temperature. Solutions of inorganic compounds are usually good conductors, whereas those of organic compounds are usually poor conductors. Conductivity is enhanced by calcium, potassium, sodium, and magnesium chlorides and sulfides. Meters can be used to measure the degree to which electrical current can travel through water. The unit of measure is 1 mS/m (millisiemens/meter) or 1 μ S/cm (microsiemens/centimeter). The reading indicates the amount of ions in the water. Although traditional chemical tests for hardness measure calcium and magnesium, they fail to provide an indication of other ions (e.g., sodium). The conductivity meter provides a much better measure of ionic strength.

ACID-VOLATILE SULFIDES

Measurement of acid-volatile sulfides (AVSs) and simultaneously extracted metal (SEM) concentrations associated with AVS extraction can provide insight into the bioavailability of metals in anaerobic (anoxic) sediments (Ankley et al., 1996; DiToro et al., 1990). AVSs are the reactive solid-phase sulfide fraction that is extracted by cold hydrochloric acid. AVSs appear to affect the bioavailability of most divalent metal ions, as the sulfide ions have a high affinity for divalent metals. This affinity results in the formation of insoluble metal sulfides with greatly reduced bioavailability. AVS concentrations in freshwater and marine sediments can range between <0.1 and >50 μ mol AVS per gram of sediment (DiToro et al., 1990). The bioavailability of metals in sediments has been predicted by comparing the molar concentration of

AVSs to the molar concentration of SEMs (methods described below). If the concentration of AVSs is greater than that of SEMs, the metals are bound in sulfide complexes with greatly limited bioavailability. However, if the concentration of AVSs is less than that of SEMs, metals may or may not be toxic due to other controlling factors (e.g., TOC). The easily extractable sulfide fraction can be measured using the acid purge and trap technique. The sample sulfide is solubilized in cold hydrochloric acid. The analytical method involves conversion of sulfides to aqueous H_2S . This may be measured with a sulfide probe or by following a wet chemistry method. In the latter method, silver sulfide is precipitated in a gas-tight assembly and flushed with nitrogen to eliminate oxidation. The precipitation is filtered, dried, and weighed. The weight is compared with the weight obtained from a non-acidified sample, and the difference is attributed to the AVS fraction (DiToro et al., 1990).

SIMULTANEOUSLY EXTRACTED METALS

A model for predicting toxicity from divalent trace metals (DiToro et al., 1990) is based on the binding of these metals to AVSs. Where the sum of the moles of the SEMs, including Ag, Cd, Cu, Ni, Pb, and Zn, is exceeded by the molar concentration of AVS, the metals are insoluble and largely unavailable to biota. The extraction of AVS and metals should be achieved using a single methodology to ensure that recoveries associated with each measure are consistent. Simultaneous extraction improves the efficiency of the methodology. SEM can be measured in filtered aliquots by atomic absorption methods (DiToro et al., 1990). Recent SEM analysis methods use inductively coupled plasma atomic emission spectrometry (Berry et al., 1999).

METALS

Low levels of trace metals occur naturally in the environment, but highly elevated levels in sediment are generally associated with human-impacted contaminant loads. Metals are partitioned in sediments as soluble free ions, soluble organic and inorganic complexes, easily exchangeable ions, precipitates of metal hydroxides, soluble organic and inorganic complexes, easily exchangeable ions, precipitates organic complexes, insoluble sulfides, and residual forms (Gambrell et al., 1976).

Current instrument methods available for the analysis of trace metals include electrochemistry (e.g., differential pulse polarography), spectrophotometry (e.g., silver diethyldithiocarbamate), atomic absorption spectrophotometry, atomic emission spectrophotometry (AES), x-ray fluorescence (XRF), and neutron activation; the most commonly used instrumental method to analyze sediments for metals is atomic absorption spectrophotometry (Puget Sound Estuary Program, 1987c). Inductively coupled plasma mass spectrometry (ICP-MS) or ICP-AES allow for simultaneous determination of many metals at sub-part-per-billion levels with little pretreatment (Berry et al., 1999; Crecelius et al., 1987).

The concentration of salt in marine or estuarine samples may interfere with metals analyses (USEPA/USACE, 1998); therefore, acid digestion and atomic absorption spectroscopy should be coupled with an appropriate technique to control for this interference. Methods in USEPA (1986b) are recommended for the analysis of

mercury in sediments, and EPRI (1986) methods are recommended for the analysis of selenium and arsenic. USEPA methods for cadmium, hexavalent chromium, copper, lead, mercury, nickel, selenium, silver, and zinc are described in USEPA (1986b). The Puget Sound Estuary Program (1987c) suggests that mercury can be extracted using vacuum distillation and analyzed by gas chromatography/mass spectrophotometry.

SYNTHETIC ORGANIC COMPOUNDS (PESTICIDES, PCBs, TCDD-DIOXIN)

Analytical techniques for measuring organic compounds require five general steps: (1) drying the sample, (2) extraction, (3) drying the extract, (4) clean up of the extract, and (5) analysis of the extract. The Puget Sound Estuary Program (1987b) recommends centrifugation or sodium sulfate to dry the sample and a solvent extraction, with application of shaker/roller, or sonication (i.e., applying sound energy). Sample drying with sodium sulfate is recommended for samples weighing approximately 10 grams (after overlying water is decanted). The sediment and sulfate mixture is extracted and the extract is processed (MacLeod et al., 1985). Soxhlet[®] extraction (USEPA, 1986b) involves distillation with a solvent such as acetone, dichloromethane/methanol (2:1), dichloromethane/methanol (9:10), or benzene/methanol (3:2). Sonication with solvent mixtures and a 30-gram subsample of sediment has been recommended (USEPA, 1983).

Drying the extract can be accomplished through separatory funnel partitioning as needed to remove water and sodium sulfate or by using a Kuderna–Danish apparatus and rotary evaporation with purified nitrogen gas for concentration to smaller volumes (Puget Sound Estuary Program, 1987c). Using the separatory funnel partitioning method, the wet sample is mixed with methanol and centrifuged. The supernatant is decanted and extracted later. Extraction of the sample is continued using less polar solvents and the water/methanol and solvent extracts are combined and dried.

Elemental sulfur can be removed from the sediment sample with vigorous mechanical agitation using a Vortex or Genie[®] or using activated copper (Puget Sound Estuary Program, 1987c). Organic interferences can be removed with gel permeation chromatography (GPC) (USEPA, 1983), bonded octadecyl columns (Puget Sound Estuary Program, 1987c), high-performance liquid chromatography (HPLC) (Metro, 1981), silica gel (Puget Sound Estuary Program, 1987c), or alumina (USEPA, 1983). Instrumental analyses for volatiles and semivolatiles and for pesticides/PCBs are performed using gas chromatography/mass spectrophotometry (GC/MS) and gas chromatography/electron capture detection (GC/ECD), respectively (Burgess and McKinney, 1996; Puget Sound Estuary Program, 1987b).

OIL AND GREASE

Oil and grease tests for sediments measure material recovered that is soluble in a nonpolar solvent under acidic conditions. Oil and grease compounds are substances such as hydrocarbons, vegetable oils, animal fats, waxes, soaps, and greases. Many solvents can dissolve other substances (e.g., sulfur compounds, organic dyes, chlorophyll). Therefore, oil and grease are operationally defined by the solvent used and the

analytical method used to perform the analysis. There are two basic methods used to analyze oil and grease: the gravimetric technique and the infrared (IR) spectrophotometer technique (Puget Sound Estuary Program, 1996).

PETROLEUM HYDROCARBONS AND PAHS

Petroleum hydrocarbons are oil and grease constituents that remain in solution after contact with silica gel. Petroleum distillates, also called hydrocarbons or petrochemicals, refer to a broad range of compounds that are extracted by distillation during the refining of crude oil. During the fractional distillation of petroleum, crude oil is heated to allow various compounds to turn from liquid into gas and is then captured as they rise, cool, and condense. Lighter, more volatile compounds rise higher before they condense and are collected on distillation trays. Heavier, less volatile compounds such as diesel fuel and oil are collected on lower distillation trays. Waxes and asphalts are collected from the bottom after the other products have volatilized.

Petroleum distillates contain both aromatic hydrocarbons (carbon rings) and aliphatic hydrocarbons (straight carbon chains). The chemical structure of the hydrocarbon largely defines the nature and behavior of these compounds. Most aromatic hydrocarbons are chronic toxins and known carcinogens. Aliphatic compounds are found in all crude oils and most petroleum products. Many aromatic hydrocarbons have a pleasant odor and include such substances as naphthalene, xylene, toluene, and benzene. Aliphatic hydrocarbons are flammable and may be explosively flammable. Aliphatic hydrocarbons include methane, propane, and kerosene.

Aromatic and aliphatic hydrocarbons were analyzed in sediments by Page et al. (1995a,b). Sediment samples were spiked with the appropriate surrogates, mixed with equal amounts of sodium sulfate to dry the samples, and extracted with a methylene chloride–acetone mixture (Method 3550, USEPA, 1986b). The concentrated extracts were partitioned on an alumina column into saturated and unsaturated hydrocarbon fractions (Method 3611, USEPA, 1986b). The fractions were concentrated using the appropriate preinjection volume, spiked with the appropriate internal standards, and analyzed by gas chromatography with flame ionization detection (GC/FID) and gas chromatography with mass spectrometry (GC/MS) detection operating in the selected ion monitoring (SIM) mode. The method of internal standards (Method 8000, USEPA, 1986b) using the average relative response factors generated from the linear initial calibration was used to quantify the target compounds. All data were corrected for the recovery of the appropriate surrogate compound. Their relative abundances could then be used for identification and quantification purposes.

Total petroleum hydrocarbons (TPHs) and polycyclic aromatic hydrocarbons (PAHs) have also been analyzed by first acidifying the sample with concentrated hydrochloric acid and then extracting hydrocarbons with a mixture of methanol and hexane. The hexane extracts were then spiked with an internal standard and analyzed by GC/FID for TPH content and by GC/MS for PAH analysis. Kaplan et al. (1996) extracted hydrocarbons using anhydrous Na_2SO_4 with methylene chloride and sonication. The total solvent extract was then concentrated with Kuderna–Danish equipment. The concentrate was further concentrated using a gentle stream of dry nitrogen. An aliquot was then injected directly into the gas chromatography.

TOTAL SULFIDES

Total sulfides represent the combined amount of acid-soluble H_2S , HS^- , and S^{2-} in a sample. Sulfides are often measured because they are common in some sediments, particularly those that are anoxic, and they can be toxic to aquatic organisms. The Puget Sound Estuary Program (1996) described a method to measure total sulfides in sediments. Oxygen is removed from the sample using nitrogen gas, methyl orange and hydrochloric acid are added, and the mixture is heated. Amine solution and iron chloride are added to develop a colorimetric reaction product, and sample absorbance is measured spectrophotometrically. Methods for measuring sulfides in aqueous samples include potentiometric methods described by ASTM (2000e) and APHA (Method 4500, 1995). Sulfide ions are measured using a sulfide ion-selective electrode in conjunction with a double-junction, sleeve-type reference electrode (Phillips et al., 1997). Potentials are read using a pH meter or a specific ion meter having a direct concentration scale for the sulfide ion. Samples are treated with a sulfide antioxidant buffer, which fixes the solution pH at a high alkaline level and retards air oxidation of sulfide ion in solution. This ensures that the sulfide measured represents total sulfides as S^- ion and rather than the HS^- or H_2S found at lower pH values. APHA (Method 4500, 1995) provides qualitative as well as quantitative methods to determine aqueous sulfide concentrations. Qualitative methods include the antimony test, silver–silver sulfide electrode test, lead acetate paper test, and silver foil test. Quantitative methods include the photometric method, automated photometric methylene blue colorimetric method, and iodometric titration method for standardizing stock solutions.

SEDIMENT OXYGEN DEMAND

Sediment can exhibit significant rates of oxygen uptake attributable to (1) a benthic ecosystem supported by soluble organic substances in the water column, (2) naturally occurring sediments derived from aquatic plants and animals, or (3) detritus discharged into the water body by natural runoff. When numerical modeling is required to predict dissolved oxygen concentrations, the rate of dissolved oxygen consumed by the benthic ecosystem is defined as the sediment (benthic) oxygen demand (SOD) in grams O_2 per m^2 per day.

Two approaches for measuring SOD were reviewed by Truax et al. (1995), including *in situ* respiratory and laboratory respirometry methods. Numerous techniques have been developed for each approach. Generally, *in situ* methods are considered more credible than laboratory measurements, although both apply the same technique. A given amount of sediment is enclosed in a chamber with a known water volume, and oxygen uptake is measured over time. The SOD rate is then calculated based on the area of the enclosed sediment, the volume of water in the chamber, and the rate of uptake.

In situ sediment oxygen demand measurement methods were described by Uchrin and Ahlert (1985). A cylindrical respirometer, dissolved oxygen probe with a stirring mechanism, and dissolved oxygen meter were used. Ambient dissolved oxygen was measured using the probe/meter as well as by using the Winkler method (APHA,

1995) in the laboratory to determine the effect of respiration on total dissolved oxygen uptake. The respirometer was deployed in a level area at the bottom of the water body. Dissolved oxygen levels were recorded initially and at 15-minute intervals thereafter to determine the SOD rate.

SEDIMENT BIOCHEMICAL OXYGEN DEMAND

Biochemical oxygen demand (BOD) is a measure of the dissolved oxygen consumed by microbial organisms while assimilating and oxidizing the organic matter in a sample (Puget Sound Estuary Program, 1996). The test is an empirical methodology in which standardized laboratory procedures are used to determine the relative oxygen uptake of environmental samples. The test measures the amount of molecular oxygen used during a specified incubation period to biochemically degrade organic material and to oxidize reduced forms of nitrogen (APHA, 1995). Plumb (1981) described a method to analyze BOD in sediments using freshwater bacteria as seed material and buffered distilled water as dilution water. The Puget Sound Estuary Program (1996) described an alternative procedure to analyze BOD in marine sediments using marine bacteria as the seed material and filtered, oxygenated seawater as the dilution water. USEPA (1987) methods should also be consulted.

SEDIMENT CHEMICAL OXYGEN DEMAND

Chemical oxygen demand (COD) is a measure of the oxygen equivalent of organic matter content in a sample that is susceptible to oxidation by a strong chemical oxidant at elevated temperature and reduced pH. The test was devised to augment the biochemical oxygen demand test. Chemical oxygen demand can be related empirically to biochemical oxygen demand, carbon, or total volatile solids. The Puget Sound Estuary Program (1996) described a method for analyzing sediment COD using a closed reflux/colorimetric method. Dichromate (Cr_2O_7) ions are used to oxidize organic matter to carbon dioxide and water and to provide oxygen. The dichromate ions remaining after the reaction are measured by titration, and the amount of oxygen consumed is then calculated. Four standard procedures for measuring COD in water are (1) open reflux method, (2) closed reflux method, (3) titrimetric method, and (4) closed reflux/colorimetric method (APHA, 1995). USEPA procedures for the colorimetric and titrimetric methods are described in USEPA (1979), and semiautomated methods are described in USEPA (1993). Hach (Loveland, CO) modified the USEPA-approved dichromate reflux method and the reactor digestion method. The methods are photometric and are adapted for use with Hach photometers.

CATION EXCHANGE CAPACITY OF SEDIMENTS

Cation exchange capacity (CEC) is a parameter that provides information relevant to metal bioavailability studies. Cations or positively charged elements (such as calcium, magnesium, hydrogen, and potassium) are attached to negatively charged surfaces of clay and organic matter. There is a continuous exchange of cations between

sediment and water. CEC is a measure of the sediment's ability to retain cationic elements. It is also a measure of clay activity and mineralogy, which are used to calculate mineralization and leaching rates and to predict interactions with contaminants. The degree of CEC is dependent on the kind and amount of suitable surfaces such as organic matter and clay. High cation exchange capacities are associated with high clay contents and high organic matter, and changes in CEC are typically associated with changes in organic carbon content and pH of the sediment. Organic matter generally supplies a greater number of exchange sites than clay particles.

REDOX POTENTIAL OF SEDIMENTS

Redox (Eh) is a measure of the oxidation–reduction potential of sediments. Measurements of Eh are particularly important for metal speciation and for determining the extent of sediment oxidation. Eh values below approximately –100 millivolts would indicate biologically important sulfide concentrations. Some trace metals form insoluble complexes with sulfides. These metal–sulfide complexes bind the metals in a form that is not bioavailable. Because free ionic metals are generally thought to possess the greatest toxicity potential, it is important to understand conditions that control binding dynamics, such as pH and Eh. Potentiometric measurements of Eh using a millivolt reader can be obtained with a platinum electrode relative to a standard hydrogen electrode (Plumb, 1981). APHA (1995) has not recommended the standard hydrogen electrode as it is fragile and impractical. Instead, their preferred method uses a silver–silver-chloride or calomel reference electrode. A graphite rather than platinum electrode for sediments is recommended (APHA, 1995). Once the Eh equilibrium is reached, the difference between the platinum or graphite electrode and the reference electrode is equal to the redox potential of the system.

TOTAL INORGANIC CARBON

Inorganic carbon has been measured as a complement to microbial activity to determine the fate of an organic contaminant in biodegradation studies (West and Gonsior, 1996) and to determine the percent carbon unaccounted for in fate transport predictions of hydrophobic contaminants (Tye et al., 1996). Often the total inorganic carbon (TIC) fraction in samples is many times greater than the TOC fraction and presents an interference in the measurement of TOC. There are several options to eliminate TIC interferences when trying to measure TOC. One option is to compensate for the IC interference by measuring total carbon (TC) and total inorganic carbon. The difference between the two is the TOC. TIC is determined by acidifying the sample to convert the inorganic carbon (i.e., carbonates, bicarbonates, and dissolved CO₂) to carbon dioxide. Carbon dioxide is purged from the sample and then detected by a nondispersive infrared detector (NDIR) calibrated to directly display the mass of carbon dioxide measured. This mass is proportional to the mass of the TIC (USEPA, 2001).

TOTAL VOLATILE SOLIDS

Total volatile solids represent the fraction of total solids that are lost on ignition at a higher temperature than that used to determine total solids. Total volatile solids are used as a crude estimate of the amount of organic matter in total solids (Puget Sound Estuary Program, 1996). In this regard, total volatile solids are often measured instead of, or in addition to, organic carbon content. Total volatile solids are operationally defined by ignition temperature. Total volatile solids content does not always represent the organic content of a sample because some organic material may be lost at the drying temperature and some inorganic material (e.g., carbonates, chlorides) may be lost at the ignition temperature. Because of the temperature dependency of total volatile solids, valid interstudy comparisons require the use of standardized drying and ignition temperatures (Puget Sound Estuary Program, 1996). Total volatile solids measurements are generally made by igniting the sediments at $550 \pm 10^\circ\text{C}$ until a constant weight is achieved and reporting the percent ash free dry weight (APHA, 1995; Keilty et al., 1988; McLeese et al., 1980).

DISSOLVED ORGANIC CARBON IN POREWATER

Dissolved organic carbon (DOC) often consists of humic substances and is the fraction of the organic carbon pool that is dissolved in water and passed through a $0.45\text{-}\mu\text{m}$ glass-fiber filter. DOC is an indicator of the chemically reactive organic fraction and accurately measures the dissolved organic load. Sediment pore waters can be rich in humic acids. As much as 50 to 90% of the porewater DOC can be colloidal, which is a significant factor because organic chemicals will preferentially partition to porewater DOC (Burgess and McKinney, 1996; Resendes et al., 1992). Hermann (1996) and Gilek et al. (1996) measured DOC using a TOC apparatus and infrared detection of CO_2 . Borgia et al. (1996) measured DOC using atomic emission spectrometry. The APHA methods for total organic carbon that can be applied to the measurement of DOC are (1) the combustion–infrared method, (2) the persulfate–ultraviolet oxidation method, and (3) the wet oxidation method (Method 5310, APHA, 1995). Adjustments for inorganic carbon interference may be required.

ALKALINITY AND HARDNESS OF THE POREWATER (FRESHWATER SEDIMENTS)

Alkalinity is defined as the acid-neutralizing (i.e., proton-accepting) capacity of water. It is the sum of all the table bases and a measure of the quality and quantity of constituents in the pore water that result in a shift in the pH toward the alkaline side of neutrality. The measured value may vary significantly with the pH endpoint used. Studies have shown that effects of certain contaminants such as metals are influenced by alkalinity as it alters speciation and bioavailability. APHA (1995) recommended a color-change titration method to measure alkalinity that was also described by ASTM (2000h). The sample is titrated with standard alkali or acid to a designated pH and the endpoint is determined electrometrically or by the color change of an internal standard. In addition, ASTM (2000b) described two additional methods:

1. A titration curve is developed to identify inflection points, a standard acid of alkali is added to the sample by small increments, and pH is recorded after each addition. The total volume of acid or alkali is plotted against the observed pH values.
2. The pH is determined and standard acid is added to lower the pH to 4.0 or less. The solution is boiled with hydrogen peroxide and titrated while hot to the phenolphthalein endpoint or, when cooled electrometrically, with standard alkali to pH 8.2, the desired endpoint.

The color-change titration method is most commonly used. Hach (Method 8202) has developed a portable water chemistry kit based on the APHA (1995) color-change titration method and an additional method using sulfuric acid with a digital titrator (Hach Method 8203). Hardness is the concentration of metallic cations, with the exception of alkali metals, present in water samples. Generally, hardness is a measure of the concentration of calcium and magnesium ions in water. Hardness is usually expressed as calcium carbonate equivalent in mg/L. Like alkalinity, hardness alters speciation and the bioavailability of certain contaminants, particularly many metals.

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