

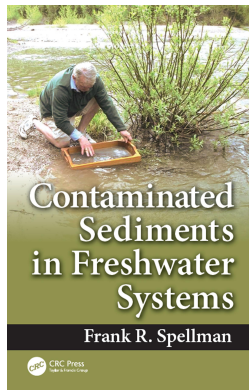
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## Contaminated Sediments in Freshwater Systems

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### Collection of Porewater

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# 13 Collection of Porewater

Sediment porewater, or interstitial water as used in this discussion, is defined as the water occupying the spaces between sediment particles. Interstitial water might occupy about 50% (or more) of the volume of a depositional (silt–clay) sediment. The interstitial water is in contact with sediment surfaces for relatively long periods of time and therefore might become contaminated due to partitioning of the contaminants from the surrounding sediments. In addition, interstitial waters might reflect groundwater–surface water transition zones in upwelling or downwelling areas. In these areas, their chemistry might be more reflective of ground or surface waters at the site. Therefore, flow, residence time, and other physicochemical factors (e.g., pH, temperature, redox potential, organic carbon, sulfides, carbonates, mineralogy) might have varying roles in determining whether interstitial waters are contaminated. Because many interstitial waters are relatively static in many depositional sediments, the contaminants in the interstitial water and in the solid phase are considered to be at thermodynamic equilibrium. This makes interstitial waters useful for assessing contaminant levels and associated toxicity. Interstitial water is often isolated to provide either a matrix for toxicity testing or an indication of the concentration or partitioning of contaminants within the sediment matrix (USEPA, 2001).

## INTERSTITIAL WATER COLLECTION AND SAMPLING

The collection of interstitial water has become increasingly important in sediment quality monitoring and remediation programs. Moreover, interstitial water sampling has become especially important in regulatory programs because interstitial water toxicity tests yield additional information not currently provided by solid-phase, elutriate (i.e., separated by washing), or sediment extract tests (Carr and Chapman, 1992; SETAC, 2001). Additionally, interstitial water toxicity tests have proved to be useful in sediment toxicity identification evaluation (TIE) studies (Burgess, 1996; Burton et al., 2003; Carr, 1998) as test procedures and sample manipulation techniques are generally less expensive, faster, and easier to conduct than solid-phase tests (SETAC, 2001). Sediment types ranging from sandy to uncompacted silt–clays are most suitable for interstitial water sampling (Sarda and Burton, 1995; SETAC, 2001). Such sampling is not typically performed on sediments with coarse particle size (such as gravel) or on hard, compacted clays, as the potential for interstitial water contamination in these sediment types is relatively low.

Note that for the purposes of discussion in this book, the principle aim is to describe sampling procedures that minimize changes in the *in situ* condition of the water. It is also important to recognize that most sediment collection and processing methods have been shown to alter interstitial water chemistry (e.g., Bufflap and Allen, 1995; Sarda and Burton, 1995; Schults et al., 1992), thereby potentially altering contamination bioavailability and toxicity.

**DID YOU KNOW?**

The Superfund program has initiated a project to develop media-specific benchmark values for those chemicals commonly found in surface water, sediment, or soil samples at sites. The values are referred to as *Ecotox Thresholds* (ETs), and they are defined as media-specific contaminant concentrations above which there is sufficient concern regarding adverse ecological effects to warrant further site investigations.

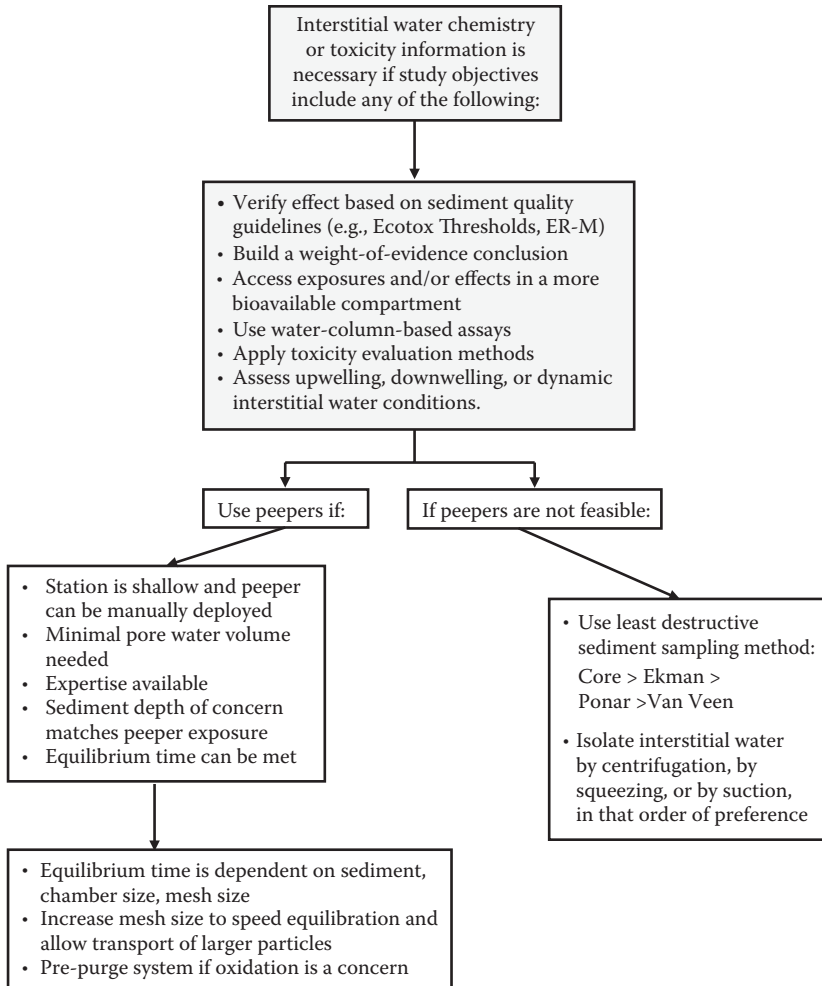
Centrifugation, pressurization, suction, and other laboratory-based methods are commonly used as alternatives to *in situ* interstitial water collection. Although these methods have been shown to alter interstitial water chemistry, they are sometimes necessary or preferred, especially when larger sample volumes are required (e.g., for toxicity testing).

As both *in situ* and laboratory-based or *ex situ* methods might be appropriate for many study objectives, it is critical to use the same procedures for all stations sampled in a study or program so appropriate sample comparisons can be made. Furthermore, the sediment depth at which interstitial water is sampled (using either *in situ* or *ex situ* extraction methods) should match the depth of interest in the study (SETAC, 2001). For example, samples for dredging remediation should be sampled to the depth to be disturbed by dredging activity, whereas samples for a status and trends survey should be collected at the biologically active depth (often <15 cm). [Figure 13.1](#) summarizes the major considerations for selecting *in situ* or *ex situ* procedures in a given study.

The two major issues of concern regarding interstitial water sample integrity are (1) the ability of the sampling device to maintain physicochemical conditions in the natural state by minimizing adsorption or leaching of chemicals to and from the device, and (2) the ability to maintain the sample in the redox state existing at the site. Precautions required to reduce the likelihood of sample artifacts will vary with each study as indicated in the following sections.

**IN SITU COLLECTION**

*In situ* methods might be superior to *ex situ* methods for collecting interstitial water, as they are less subject to sampling/extraction related artifacts and therefore might be more likely to maintain the chemical integrity of the sample (ASTM, 2000a; Sarda and Burton, 1995; SETAC, 2001). However, *in situ* methods have generally produced relatively small volumes of interstitial water and are often limited to wadeable or diver-accessible water depths. These logistical constraints have limited their use and applicability in sediment monitoring studies. The principal methods for *in situ* collection of interstitial water involve either deploying peepers (Adams, 1991; Bottomly and Bayly, 1984; Brumbaugh et al., 1994; Bufflap and Allen, 1995; Carignan and Lean, 1991; Carignan et al., 1985) or suction techniques (Howes et al., 1985; Knezovich and Harrison, 1988; Watson and Frickers, 1990). A summary



**FIGURE 13.1** Considerations for selecting the appropriate type of interstitial water sampling method.

of these methods is provided in [Table 13.1](#). Both methods have a high likelihood of maintaining *in situ* conditions. In cases where *in situ* deployment is impractical, peepers or suction device can be placed in relatively undisturbed sediments collected by core or grab samplers.

### PEEPER METHODS

Peepers are small chambers with membrane or mesh walls containing either distilled water or clean water of the appropriate salinity or hardness. Samples are collected by burying the devices in sediments and allowing surrounding interstitial waters to infiltrate. In principle, dissolved solutes will diffuse through the porous wall into

**TABLE 13.1**  
***In Situ* Interstitial Water Collection Methods**

Device	Sediment Depth (cm)	Sample Volume (L <sup>3</sup> )	Advantages	Disadvantages
Peeper	0.2–10	≤0.5	Most accurate method, reduced artifacts, no lab processing; relatively free of effects from temperature, oxidation, and pressure; inexpensive and easy to construct; some selectivity possible, depending on the nature of sample, via specific membranes; wide range of membrane/mesh pore sizes and internal solutes or substrates available	Requires deployment by hand, thus requiring diving in water >0.6 m in depth; requires hours to days for equilibration (varies with site and chamber); methods are not standardized and are used infrequently; some membranes, such as dialysis/cellulose, are subject to biofouling; must deoxygenate chamber and materials to prevent oxidation effects; some construction materials yield chemical artifacts; some chambers only allow small sample volumes; care must be used on collection to prevent sample oxidation
<i>In situ</i> suction	0.2–30	≤0.25	Reduced artifacts, gradient definition; rapid collection; no lab processing; closed system, which prevents contamination; methods include airstone, syringes, probes, and core-type samplers	Requires custom, nonstandard collection devices; small volumes; limited to softer sediments; core airstone method; difficult in some sediments and in deeper water (>1 m); method might require diving for deployment in deep waters; methods used infrequently and by limited number of laboratories

*Sources:* Adapted from Sarda and Burton (1995) and SETAC (2001).

*Note:* Incorporation of filtration into any collection method might result in loss of metal and organic compounds.

the peeper, and the contained water will reach equilibrium with the ambient interstitial water. The design concept for sediment peepers originated as modifications of the dialysis bag technique used by Mayer (1976) and Hesslein (1976), and it has been modified successfully for use in laboratory sediment toxicity tests (Doig and Liber, 2000). The initial designs consisted of either a flat base plate or a cylindrical dialysis probe (Bottomley and Bayly, 1984) with compartments covered by dialysis membranes and a manifold for collection of multiple samples at various depths in the sediment profile. Further modifications to these designs have incorporated sampling ports, large sample compartments, and various types of membranes with different port sizes. These modifications are usually required based on specific project objectives regarding sample volumes and contaminants of interest.

Various peeper devices have been used effectively to collect interstitial water. For example, a simplified design using a 1- $\mu\text{m}$  polycarbonate membrane over the opening of a polyethylene vial was successful in capturing elevated levels of copper and zinc (Brumbaugh et al., 1994). Other designs have been used to collect nonpolar organic compounds in a variety of aquatic systems (Axelman et al., 1999; Bennett et al., 1996) and in overlying water (Huckins et al., 1990). Peepers have also been used to expose organism to sediments *in situ* (Burton et al., 2003). Burton et al. (1999) successfully introduced organisms to aerobic sediments using peepers; however, anoxic sediments are not amenable to *in situ* organism exposure.

The use of various materials might be advisable when constructing peepers, depending on the contaminants of concern; for example, for many contaminants, peepers constructed from acrylic materials appear to yield interstitial water samples with minimal chemical artifacts (Burton et al., 2003). Some polymer materials might be inappropriate for studies of certain nonpolar organic compounds. Cellulose membranes are also unsuitable, as they decompose too quickly. Plastic samplers can contaminate anoxic sediments with diffusible oxygen (Carignan et al., 1994).

In preparation for interstitial water collection, peeper chambers should be filled with deoxygenated water, which can be prepared by nitrogen purging for 24 hours prior to insertion. If sediment oxidation is a concern, the peepers should be transported to the deployment site in a sealed oxygen-free water bath to avoid potential changes to the sediment water equilibrium caused by dissolved oxygen interactions. However, during peeper equilibration periods, anoxic conditions are likely to be quickly reestablished. In addition, when samples are collected and processed, exposure to oxygen should be minimized. Following initial placement, the equilibrium time for peepers may range from hours to a month, but a deployment period of 1 to 2 weeks is most often used (Adams, 1991; Call et al., 1999; Steward and Malley, 1999). Equilibration time is a function of sediment type, study objectives, contaminants of concern, and temperature (e.g., Carr et al., 1989; Howes et al., 1985; Mayer, 1976; Simon et al., 1985; Skalski and Burton, 1991). Membrane pore size also affects equilibration time, with larger pore sizes being used to achieve reduced equilibration times (Sarda and Burton, 1995). For example, using a peeper with a 149- $\mu\text{m}$  pore size, Adams (1991) reported equilibration of conductivity within hours of peeper insertion into the sediment. Thus, it appears that equilibration time is a function of the type of contaminant, sediment type, peeper volume, and mesh pore size.

Peepers with large-pored membranes shorten equilibration times and allow particulates to enter the chamber. The larger solids tend to settle to the bottom of the peeper chamber, and caution should be used to avoid collecting the solids when retrieving the water sample from the chamber. Colloidal particles will remain suspended in the sample and thereby present an artifact, but the concentration of such particles is typically lower than that found in laboratory-centrifuged samples (Chin and Gschwend, 1991).

## SUCTION METHODS

Various suction devices are available for collecting interstitial water. A typical suction device consists of a syringe or tube of varying length, with one or more ports located at the desired sampling positions (ASTM, 2000a). The device is inserted

**DID YOU KNOW?**

The potential for high variability in interstitial water chemical characteristics should be taken into account when developing the sampling design. This is the case because several studies and analyses of interstitial water from replicate peepers have demonstrated from low to high heterogeneity in water quality characteristics (Frazier et al., 1996; Sarda and Burton, 1995).

into the sediment to the desired depth and a manual, spring-operated, or vacuum gas suction is applied to directly retrieve the water sample. A variation on this approach employs a peeper-like porous cup or perforated tube with filters. The unit is inserted into the sediment for a period of time, allowing interstitial water to infiltrate the chamber before suction is applied. The samples are then retrieved by suction. Another variation that has been used successfully employs an airstone embedded into the sediment which forces interstitial water upward where it can be collected via syringe or tube. All of these suction methods generally yield smaller quantities of interstitial water than peepers, and chemical (toxicological) artifacts are more likely due to greater potential exposure of interstitial water to oxygen (ASTM, 2000a).

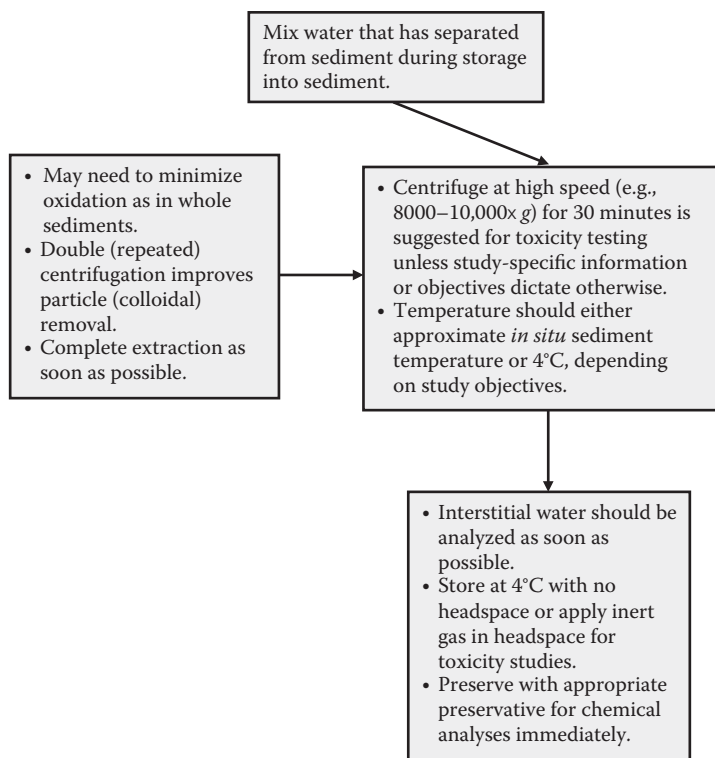
**PROCESSING OF FIELD-COLLECTED INTERSTITIAL WATER SAMPLES**

Following sample retrieval, interstitial water might have to be recovered and stabilized quickly to prevent oxidative changes or volatilization (Carignan, 1984). Containers should be filled, with no headspace, to minimize changes in dissolved oxygen and contaminant bioavailability. Procedures for stabilization are dependent on the analyses to be performed. When nonvolatile compounds are the target analytes, acidification is often stipulated, whereas organic carbon and methane may be stabilized with saturated mercury chloride (Mudroch and MacKnight, 1994). Samples to be analyzed for toxicity are normally cooled to 4°C as soon as possible for transport to the laboratory. U.S. Environmental Protection Agency (USEPA) methods for toxicity testing of surface waters and effluents recommend that samples not be frozen in storage or transport (USEPA, 1991). However, later information suggests that freezing of interstitial water may not affect toxicity in some cases (Carr and Chapman, 1995; Ho et al., 1997; SETAC, 2001). Unless a demonstration of acceptability is made for the sites of interest, interstitial water samples should not be frozen prior to biological testing. Samples for chemical analyses should be preserved immediately, if appropriate, or cooled to 4°C as soon as possible.

**EX SITU EXTRACTION OF INTERSTITIAL WATER**

*Ex-situ* interstitial water collection methods are often necessary when relatively large volumes of interstitial water are required (such as for toxicity testing), when *in situ* collection is not viable, or when a brief sampling time is critical. Although these extraction methods can be done in the field or in the laboratory, extraction in





**FIGURE 13.2** Summary of recommended procedures and considerations for laboratory isolation of interstitial water.

the laboratory just prior to analysis or testing is preferable so the sample is maintained as close to its original state as much as possible during transport and storage (SETAC, 2001). Guidance in this section reflects recommendations presented in several publications, including proceedings from workshops devoted entirely to interstitial water extraction methods, water handling, and their use in toxicity applications: (1) a dredged materials management program workshop on interstitial water extraction methods and sample storage in relation to tributyltin analysis (Hoffman, 1998), and (2) a Pellston workshop on interstitial water toxicity testing, including interstitial water extraction methods and applications (SETAC, 2001). [Figure 13.2](#) summarizes many of the issues associated with laboratory isolation of interstitial water discussed in this section.

### DID YOU KNOW?

Emphasis should be placed on minimizing the duration of all sample manipulations whenever possible.



Centrifugation and squeezing are the two most common techniques for collecting interstitial water, and they are generally preferred when large volumes are required. Other methods include pressurization (e.g., vacuum filtration) devices, which can be used to recover small volumes of interstitial water. Regardless of the method used, interstitial water should be preserved immediately for chemical analyses, if appropriate, or analyzed as soon as possible after sample collection if unpreserved (such as for toxicity testing) (Hoffman, 1998; SETAC, 2001). Significant chemical changes can occur even when interstitial water is stored for periods as short as 24 hours (Hulbert and Brindle, 1975; Kemble et al., 1999; Sarda and Burton, 1995; SETAC, 2001; Watson et al., 1985).

If sediments are anoxic, as most depositional sediments are, sample processing, including mixing of interstitial water that has separated from the sediment, should be conducted in an inert atmosphere or with minimal atmospheric contact. Exposure to air can result in oxidation of contaminants, thereby altering bioavailability (Bray et al., 1973; Howes et al., 1985; Lyons et al., 1979). Air exposure can also result in loss of volatile sulfides, which might increase the availability of sulfide-bound metals (Allen et al., 1993; Bufflap and Allen, 1995). In addition, iron and manganese oxyhydroxides are quickly formed upon exposure to air. These compounds readily complex with trace metals, thus altering metals-related toxicity (Bray et al., 1973; Bufflap and Allen, 1995; Burton, 1991; Troup et al., 1974). Maintaining anoxic processing conditions is not necessary when study objectives are concerned with exposures to aerobic sediments, or if target contaminants are unaffected by oxidation in short-term toxicity or bioaccumulation testing.

As just mentioned and repeated here for emphasis, interstitial water filtration should be avoided (SETAC, 2001). Numerous studies have shown that filters reduce toxicity and contaminant concentrations by retaining contaminant-associated particles and also by contaminant sorption onto the filter matrix (Bray et al., 1973; Sasson-Brickson and Burton, 1991; Schults et al., 1992; Troup et al., 1974). If filtration is stipulated by a test method, treated filters (e.g., pre-soaked in distilled, deionized water, or combusted at 400°C overnight for glass-fiber filters) should be used, and unfiltered samples should also be tested for toxicity and contaminant concentrations. The characteristics of filters and the filtering apparatus should also be carefully considered, as different filters have different sorptive capacities for different contaminants.

#### **DID YOU KNOW?**

Filtration should be avoided unless required by a test method because it might reduce interstitial water toxicity. Double (serial) centrifugation (low speed followed by high speed) should be used instead. If filtering is required by a test method, pretreated filters should be used to reduce potential contamination (SETAC, 2001).

## CENTRIFUGATION

Centrifugation is the generally preferred laboratory method for collection of interstitial water (SETAC, 2001). It is a relatively simple procedure that allows rapid collection of large volumes of interstitial water. It also facilitates the maintenance of anoxic conditions (if required). However, centrifugation, like other *ex situ* procedures, might yield chemical or toxicology artifacts due to the extraction procedures themselves, which might alter the natural equilibrium between interstitial water and sediment.

Prior to centrifugation, the sediment sample is homogenized (i.e., complete mixing of the sediment to obtain a consistency of physicochemical properties throughout the sample) and partitioned among centrifuge bottles. If the homogenized sample is stored prior to centrifugation, interstitial water might accumulate on the surface of the sediment. This overlying water should be mixed into the sediment before subsampling for centrifugation. Samples are then partitioned among centrifuge bottles. In general, approximately 50% of sediment moisture content can be extracted as interstitial water. If interstitial water volume requirements are lower, smaller sediment subsamples may be used.

Based on research to date, both slower and faster centrifugation speeds (and associated differences in colloid/suspended solids removal) may be appropriate depending on the study objectives. For many programs that are interested in characterizing site toxicity, high-speed centrifugation may not be appropriate because one is interested in the toxicity potential of the interstitial water in its entirety (i.e., including colloidal material). However, if one is interested in comparing interstitial water contaminant concentration to specific sediment quality values or model exposure compartments, for example, then high-speed centrifugation might be necessary. As our knowledge is still limited in this area, it is perhaps most important to note that centrifugation speed often has a dramatic effect on observed sample toxicity and chemical characteristics. Therefore, in any sediment monitoring study, one centrifugation protocol (including speed and time) should be identified and used throughout for all samples.

Centrifugation has been performed at various temperatures. ASTM (2000a) recommended that the centrifugation temperature reflect the *in situ* sediment temperature to ensure that the equilibrium between the particulate and interstitial water is not altered. Alternatively, a temperature of 4°C may be preferred to minimize temperature-mediated chemical and biological processes (Environment Canada, 1994). When centrifuging coarse sand, it might be desirable to use a modified centrifuge bottle to aid interstitial water recovery (USEPA/USACE, 1998). The modified bottle is equipped with an internal filter that can recover 75% of the interstitial water, as compared to 25 to 30% recovery from squeezing (Saager et al., 1990).

As discussed in Chapter 12, all containers have limitations with regard to adsorption or leaching of chemicals, ease of use, and reliability. For example, polytetrafluororthylene (PTF) bottles have been used successfully up to 2500× *g* when filled to 80% of capacity but collapse at 3000× *g* (Burgess et al., 1993). Polycarbonate bottles have been used successfully for tributyltin analyses in interstitial water (Hoffman, 1998). If small volumes of water are required for testing, higher speed centrifugation can be performed with glass tubes (up to 10,000× *g*) (Word et al., 1987). Larger glass

tubes, however, cannot be centrifuged at such high speeds. If metal toxicity is not a concern, then high-speed centrifugation in larger stainless steel centrifuge tubes is suitable. If test samples are contaminated with photoreactive compounds such as polycyclic aromatic hydrocarbons (PAHs), exposure of the sample to light should be minimized to limit degradation or alteration of potentially toxic compounds. This can be accomplished by using reduced lighting.

### SEDIMENT SQUEEZING

Isolation of interstitial water by squeezing has been performed using a variety of procedures and devices (Adams, 1991; Carr, 1998; Carr and Chapman, 1992; Carr et al., 1989; Jahnke, 1988; Kalil and Goldhaker, 1973; Long et al., 1990; Reeburgh, 1967; Watson and Frickers, 1990). Inexpensive, low-pressure mechanical squeezers can be constructed that may provide specialized capacities such as collection of interstitial water profiles from core samples (Bender et al., 1987). In all cases, the interstitial water is passed through a filter that is a part of the squeezing apparatus.

Squeezing has been shown to produce a number of artifacts due to shifts in equilibrium from pressure, temperature, and gradient changes (e.g., Bollinger et al., 1992; Froelich et al., 1979; Kriukov and Manheim, 1982; Schults, 1992). Squeezing can affect the electrolyte concentration in the interstitial water particularly with a decrease in chemical concentration in the interstitial water near the end of the squeezing process. However, others have reported that squeezing did not produce artifacts in interstitial water toxicity studies (Carr, 1998; Carr and Chapman, 1995; SETAC, 2001). It is therefore recommended that, if squeezing is performed, moderate pressures be applied along with electrolyte (conductivity) monitoring during extraction (Kriukov and Manheim, 1982). Squeezing should also be performed at *in situ* ambient temperatures, as significant alterations to interstitial water composition can occur when squeezing is conducted at temperatures different from ambient conditions (e.g., Bischoff et al., 1970; Mangelsdorf et al., 1969; Sayles et al., 1973).

Other sources of interstitial water alteration during squeezing are contamination from overlying water, internal mixing of interstitial water during extrusion, and solid-solution reactions as interstitial water is expressed through the overlying sediment. As interstitial waters are displaced into upper sediment zones, they come in contact with solids with which they are not in equilibrium. This intermixing causes solid-solution reactions to occur. Most interstitial water chemical species are rapidly transformed, as observed with ammonia and trace metals (Rosenfield, 1979; Santschi et al., 1997). Bollinger et al. (1992) found elevated levels of several ions and dissolved organic carbon in squeezed samples as compared to samples collected by *in situ* peepers. The magnitude of the artifact will depend on the pollutant sediment characteristics and redox potential.

### PRESSURIZED AND VACUUM DEVICES

Other methods for extraction of interstitial water from sediment samples can include vacuum filtration (Jenne and Zachara, 1987; Knezovich and Harrison, 1987; Winger and Lasier, 1991), gas pressurization (Reeburgh, 1967), and displacement (Adams,

1991). These methods typically recover only small volumes of interstitial water and are not commonly used. Use of a hand vacuum with an aquarium stone is an effective vacuum filtration method (Sarda and Burton, 1995; Winger and Lasier, 1991). The procedure typically involves attaching the air stone to a 50-mL syringe via plastic tubing, inserting it into the sediment to the desired depth, and then applying suction. This method can recover relatively large volumes of interstitial water. Santschi et al. (1997) used this procedure to extract up to 1500 mL from 4 L of sediment. Sarda and Burton (1995) found that ammonia concentrations in water obtained by this procedure were similar to those collected by *in situ* peepers. Drawbacks to this method include loss of equilibrium between the interstitial water and the solids, filter clogging, and oxidation (Brinkman et al., 1982).

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