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Region 4, Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Pore Water Sampling**

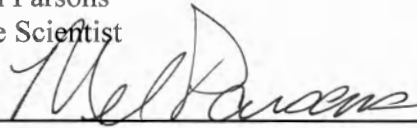
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1 General Information

1.1 Purpose

The purpose of this operating procedure is to describe the procedures, methods and considerations to be used when obtaining a sediment pore water sample.

1.2 Scope/Application

This document describes procedures generic to all pore water sampling methods to be used by field personnel when collecting and handling samples in the field. On the occasion that Science and Ecosystem Support Division (SESD) personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a pore water sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version.

M.H.E. Products. 2003. PushPoint Sampler (US Pat. # 6,470,967) Operators Manual and Applications Guide, Version 2.01. East Tawas, MI. <http://www.mheproducts.com>

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version.

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version.

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version.

SESD Operating Procedure for Surface Water Sampling, SESDPROC-201, Most Recent Version.

SESD Operating Procedure for Pump Operation, SESDPROC-203, Most Recent Version.

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version.

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version.

SESD Operating Procedure for Groundwater Sampling, SESDPROC-301, Most Recent Version.

SESD Operating Procedure for Potable Water Supply Sampling, SESDPROC-305, Most Recent Version.

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version.

USEPA. ASBLOQAM. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4, Science and Ecosystem Support Division, Athens, GA. Most Recent Version.

USEPA. SHEMP. Safety, Health and Environmental Management Program Procedures and Policy Manual. Science and Ecosystem Support Division, Region 4, Athens, GA. Most Recent Version.

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version.

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting pore water samples. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual (most recent version) and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting pore water samples:

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook. Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as bills of lading, will be retained by the project leader and stored in a secure place.

1.5.3 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-002 (most recent version). Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation, in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 (most recent version), and SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005 (most recent version).

2 Sampling Methodology

2.1 General

The pore water sampling techniques and equipment described in this procedure are designed to minimize effects on the chemical and physical integrity of the sample. If the procedures in this section are followed, a representative sample of the pore water should be obtained.

2.2 Collection Considerations

The physical location of the investigator when collecting a sample may dictate the equipment to be used. Wading is the preferred method for reaching the sampling location, particularly if the stream has a noticeable current (i.e., is not impounded). However, wading may disrupt bottom sediments causing biased results; therefore, the sampler should enter the area downstream of the sampling location and collect sample facing upstream. If the stream is too deep to wade, the pore water sample may be collected from a platform such as a boat or by SCUBA diving. If sampling from a boat or in water deeper than the length of the sampler, extensions may be utilized. The device is suitable for use only in fine-grained material (no gravel or cobble).

2.3 Summary of Procedure

Sediment pore water is collected using a pore water extracting device (Figure 1). The most common type used is the PushPoint™ sampler (M.H.E. Products 2003), made out of stainless steel tubing. The sampling end of the pore water device is inserted into the sediment to the desired depth, and pore water is extracted using a syringe or peristaltic pump. Other similar devices may be used providing that the integrity of the sample is maintained and no ambient surface water is allowed in contact with the sample.

2.4 Sampling Equipment

A PushPoint™ or similar sampler typically consists of a pointed tubular stainless steel tube with a screened zone at one end and a sampling port at the other. The pointed end with the screened zone consists of a series of very fine interlaced machined slots to allow pore water to enter the sampler. A removable guard rod adds rigidity to the sampler during sediment insertion. The length of the screened zone will depend on the site specific study design. Depending on the data quality objectives (DQO) of the study, filters may be placed over the screened zone if additional screening is needed. Pore water is collected through the opposite end of the device by connecting flexible tubing and using a syringe or peristaltic pump to extract the sample. Teflon® tubing is the preferred tubing to be used for collecting pore water samples. However, other tubing may be used, depending upon the DQOs for the specific application.

There are many modifications that can be incorporated into the procedure to satisfy data quality objectives for a specific application. The procedures discussed in the following sections provide guidance on the basic operation of pore water sampling devices and issues to consider when collecting pore water.

An alternative system is available in SESD inventory for use in soft sediments in water deeper than wading depth. A well screen and short riser approximately ¾” in diameter has external threads to fasten to the bottom of a custom flange and internal threads to accept a tubing compression adapter. The accompanying rimmed flange has a coupling with both top and bottom threads. The well screen is screwed into the bottom of the flange coupling and Teflon® tubing is attached to the tubing compression adapter which is threaded into the well screen. The tubing is then inserted through the pipe or well casing which is then screwed into the upper coupling threads. The entire assembly can be deployed in water up to ten feet of depth from a well anchored boat.

2.5 Pore Water Sampler Deployment Considerations

It is critical in the collection of pore water to avoid surface water intrusion. Water will flow in a path of least resistance. If space is created around the sides of the sampling end of the pore water device during deployment, surface water may flow down the outside of the device to the screened area and into the intended sample. Therefore, the pore water device should be used with a sampling flange (Figure 2), especially when collecting pore water near the sediment-surface water interface. If pore water is collected from deep in the sediments, a flange may not be necessary. When inserted through the sampling platform, or flange, the body of the pore water device should form a water tight seal to eliminate surface water intrusion during sample collection. Flanges should include a cutting ring to enhance sealing. Flange systems can be augmented by flexible plastic sheeting of appropriate material. The sheeting can be weighted to conform to the stream bottom by sediments obtained from other areas of the stream or banks away from the sampling location.

The flange can be made of any material that will not cross contaminate the intended sample. If both inorganic and organic analyses are required, the flange should be made of inert material such as stainless steel or Teflon®. The size of the flange depends on the volume of pore water to be collected. If large volumes of pore water are to be collected, use a large flange size. A useful estimate can be made for planning by taking the entire required water volume, tripling it to assume 33% porosity, and then calculating the dimensions of a sphere or cylinder of this volume. The flange should cover at least this estimated volume. If it is not practical to use a large flange, then multiple devices may be deployed and smaller volumes can be collected from several devices for a composite sample. If multiple devices are deployed, they should be spaced an appropriate distance apart so they will not interfere with one another.

For irregular surfaces a flange can be improvised from polyethylene sheeting weighted by shovelled nearby stream sediments. Several of the flanges in SESD inventory have a

threaded nut and washer to facilitate sealing the flange to a polyethylene sheet for this purpose.

In general, the volume of pore water that can be collected at a given location is limited. Collecting large volumes of pore water will ultimately result in the collection of water from the overlying water body. Often, minimum required volumes must be negotiated with the laboratory to limit the volumes withdrawn.

Where significant differences in parameters such as pH or conductivity exist between the surface water and pore water, a check can be made at the end of sampling to assess whether surface water intrusion has occurred by measuring the pore water parameters at the beginning and conclusion of sampling.

2.6 Pore Water Collection

The flange is first placed at the desired sampling point with the push-point removed to allow water to escape from under the flange. The flange rim should be carefully worked into the sediment until the flange is flush with the sediment surface. The pore water device should then be inserted through the compression adapter on the flange and into the sediment as carefully as possible (Figure 2). When the sampler is inserted to the desired depth, the compression adapter should be tightened. The push-point's guard rod can then be withdrawn. Do not reinsert the guard rod into the sampler for any reason until the sampler has been cleaned (sediment particles rolled between the two metal surfaces will lock the parts together and permanently damage the sampler.)

When deploying the pore water device, care must be taken not to disturb the sampling area. If the sampler is wading in the water body, the sampler should lean out and insert the pore water device as far as possible away from where the sampler is standing to reduce potential effects of the sampler on the integrity of the pore water sample. Depth of penetration of the pore water device into the sediment depends on the objectives of the specific investigation.

After the pore water device has been successfully deployed, attach the sample tubing to the sampling port of the pore water device. Short pieces of Silastic® tubing can be used to splice Teflon® sample tubing to a push-point sampler, taking care to butt the tubing to the sampler at the center of the splice. Then attach the other end of the tubing to a sample withdrawing device, such as a syringe or a peristaltic pump (according to SESD Operating Procedure for Pump Operation, SESDPROC-203). Before collecting a pore water sample, be sure to purge out all air and surface water from the pore water device and sample tubing with the appropriate amount of pore water. If utilizing a syringe for collection, a three-way valve with a side syringe must be utilized for the surface water purge in order not to cross contaminate the sampling syringe.

2.6.1 Peristaltic Pump/Vacuum Jug Collection

The peristaltic pump/vacuum jug can be used for sample collection of organic or inorganic samples because it allows for the sample to be collected without coming in contact with the pump head tubing, maintaining the integrity of the sample. This is accomplished by placing a Teflon® transfer cap assembly onto the neck of a pre-cleaned standard 1-liter amber glass container (Figure 3). Teflon® tubing (¼-inch O.D.) connects the container to both the pump and the sample source. The pump creates a vacuum in the container, thereby drawing the sample into the container without it coming into contact with the pump head tubing.

Because the sample is exposed to a vacuum and is agitated as it enters the vacuum jug, this method cannot be used for collection of samples for volatile organic compounds. An alternative method for collecting volatile organics involves filling the Teflon® tubing with sample by running the pump for a short period of time. Once the tubing is full of water, the tubing is removed from the pore water sampler and, then pinched off at the pump in order to maintain the vacuum and disconnected from the pump head tubing. The water is then allowed to carefully drain, by gravity, into the sample vials. Alternatively, without disconnecting the tubing from the pump head, the contained sample can be pushed out of the tubing, into the sample vials, by reversing the peristaltic pump at very low speed. Great care must still be taken with this method in order not to agitate the sample during the transfer process or transfer water that has been in contact with the Silastic® tubing into the vials.

Because pore water is typically collected from an anaerobic environment, it is preferable, especially when collecting samples for nutrient analysis, to maintain the integrity of the sample by minimizing exposure to air. This can be accomplished by purging the sample container with an inert gas such as nitrogen or argon prior to sampling. In addition, if analyzing for nutrients or metals, the container can be pre-preserved in order to minimize exposure of the sample to ambient conditions.

An alternative, when collecting samples for metals, nutrients or other sample analysis not affected by the Silastic® tubing and exposure to air is not a concern, is to collect the sample directly from the discharge of the pump head tubing after an adequate purge has been demonstrated. When collecting samples in this manner, there are several considerations to be aware of. The pump head tubing (Silastic®, etc.) must be changed after each sample and a rinsate blank must be collected from a representative piece of the pump head tubing (only one blank per investigation). Also, precautions must be taken to ensure that the end of the discharge tubing is not allowed to touch the ground or other surface to ensure the integrity of the sample collected in this manner.

2.6.2 Syringe

An alternative to using the pump and vacuum container is to use a syringe as the mechanism to draw the pore water through the sampling device. The tubing from the sampling port of the pore water device can be directly attached to a syringe with a three-way valve and a side syringe and the pore water sample can be manually withdrawn from the sediment. The valve is first switched to the side syringe, which is used for purging air and any ambient surface water in the system prior to sampling. The volume to be purged is determined by the length and diameter of the sampling device and attached tubing. Once the sampler has been purged, the valve is switched to the sampling syringe and the sample is drawn into the syringe. The syringe can be used as the final sample container or the pore water can be transferred to another container, depending on project objectives and analytical requirements.

2.7 Quality Control

If possible, a control or background sample should be collected from a location not affected by the possible contaminants of concern and submitted with the other samples. In streams or other bodies of moving water, the control sample should be collected upstream of the sampled area. For impounded bodies of water, particularly small lakes or ponds, it may be difficult or inappropriate to obtain an unbiased control from the same body of water from which the samples are collected. In these cases, it may be appropriate to collect a background sample from a similar impoundment located near the sampled body of water if there is a reasonable certainty that the background location has not been impacted. Equipment blanks should be collected if equipment is field cleaned and reused on-site or, if necessary, to document that low-level contaminants were not introduced by pumps, bailers or other sampling equipment.

2.8 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect pore water samples shall be cleaned as outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205 (most recent version) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206 (most recent version) and repaired, if necessary, before being stored at the conclusion of field studies. Cleaning procedures utilized in the field or field repairs shall be thoroughly documented in field records.

3 Special Sampling Considerations

3.1 Volatile Organic Compounds (VOC)

Pore water samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two week holding time, whereas, unpreserved samples have only a seven day holding time. During most sampling events, preserved vials are used due to their extended holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

Samples for VOC analysis must be collected using either stainless steel or Teflon® equipment. Samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be refilled. Care should be taken not to flush any preservative out of the vial during topping off. If, after attempting to refill and cap the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

3.2 Dissolved Metals Sample Collection

If a dissolved metals pore water sample is to be collected, an in-line filtration should be used. The use of disposable, high-capacity filter cartridges (barrel-type) or membrane filters in an in-line filter apparatus is preferred. The high-capacity, barrel-type filter is preferred due to the higher surface area associated with this configuration.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations; these include filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloiddally-associated trace elements, and concentration of organic matter. Therefore, consistency is critical in the comparison of short-term and long-term results. Further guidance on filtration may be obtained from Section 4.7.3 of the SESD Groundwater Sampling Procedure (SESDPROC-301).

3.3 Special Precautions for Pore Water Sampling

- A clean pair of new, non-powdered, disposable latex gloves will be worn each time a different location is sampled and the gloves should be donned prior to handling sampling equipment and sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- All background or control samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Samplers must use new, verified, certified clean disposable equipment, or pre-cleaned non-disposable equipment. Non-disposable equipment should be pre-cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205), for collection of samples for trace metals or organic compound analyses.

3.4 Sample Handling and Preservation Requirements

1. Pore water will typically be collected from sediments using a peristaltic pump and placed directly into the sampling containers. In some cases a syringe may be used to collect the sediment pore water and then transfer the sample into the appropriate container.
2. During sample collection, if transferring the sample from a collection device, make sure that the device does not come in contact with the sample containers.
3. Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 3.1).
4. All samples requiring preservation must be preserved as soon as practically possible, soon after sample collection. If pre-preserved VOA vials are used, these will be preserved with concentrated hydrochloric acid prior to departure for the field investigation. For all other chemical preservatives, SESD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not acceptably preserved, additional preservative should be added to achieve adequate preservation. Preservation requirements for surface water samples are found in the USEPA Analytical Support Branch *Laboratory Operations and Quality Assurance Manual* (USEPA ASBLOQAM).

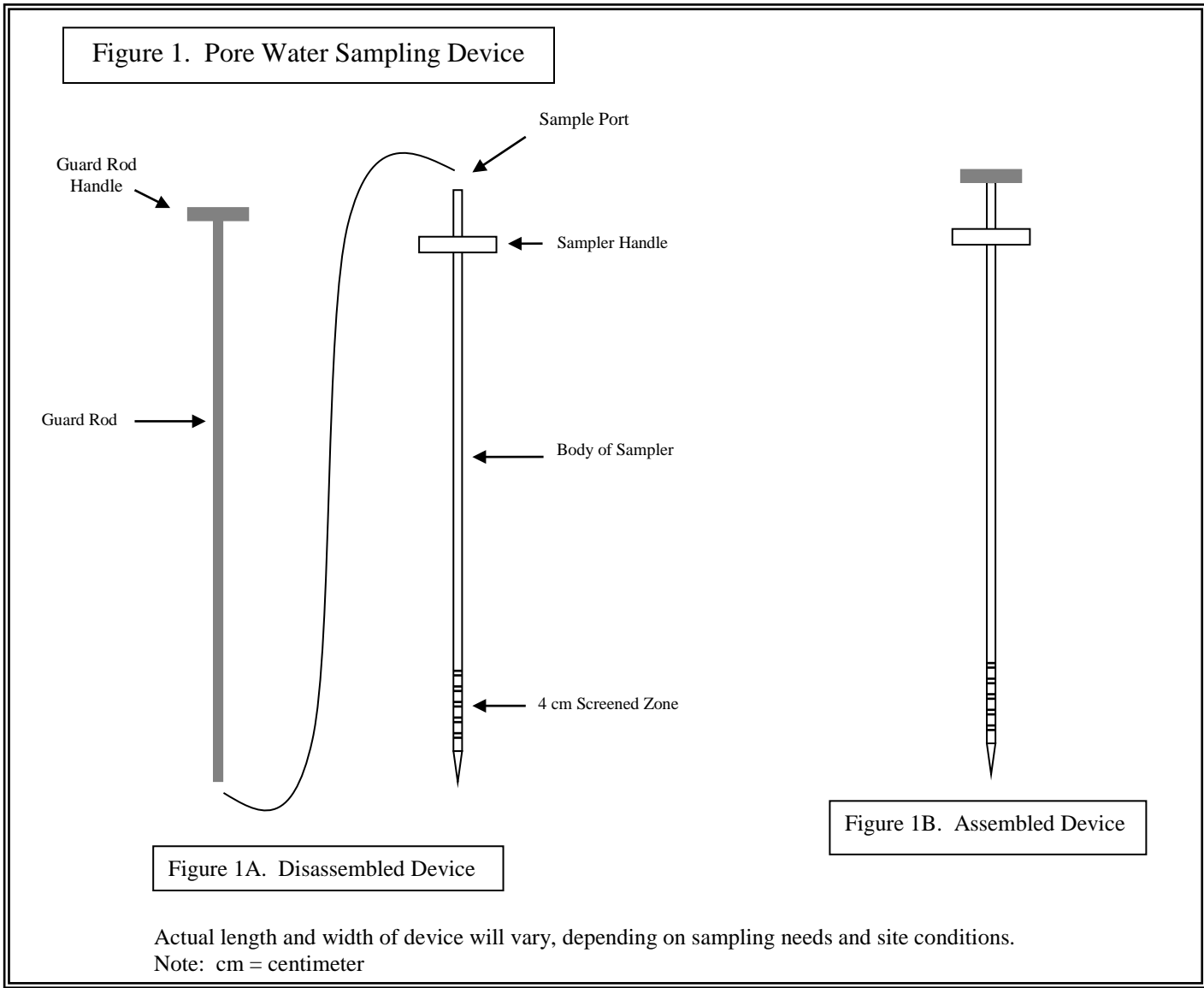


Figure 2. Pore Water Sampler Deployment Using a Sampling Flange

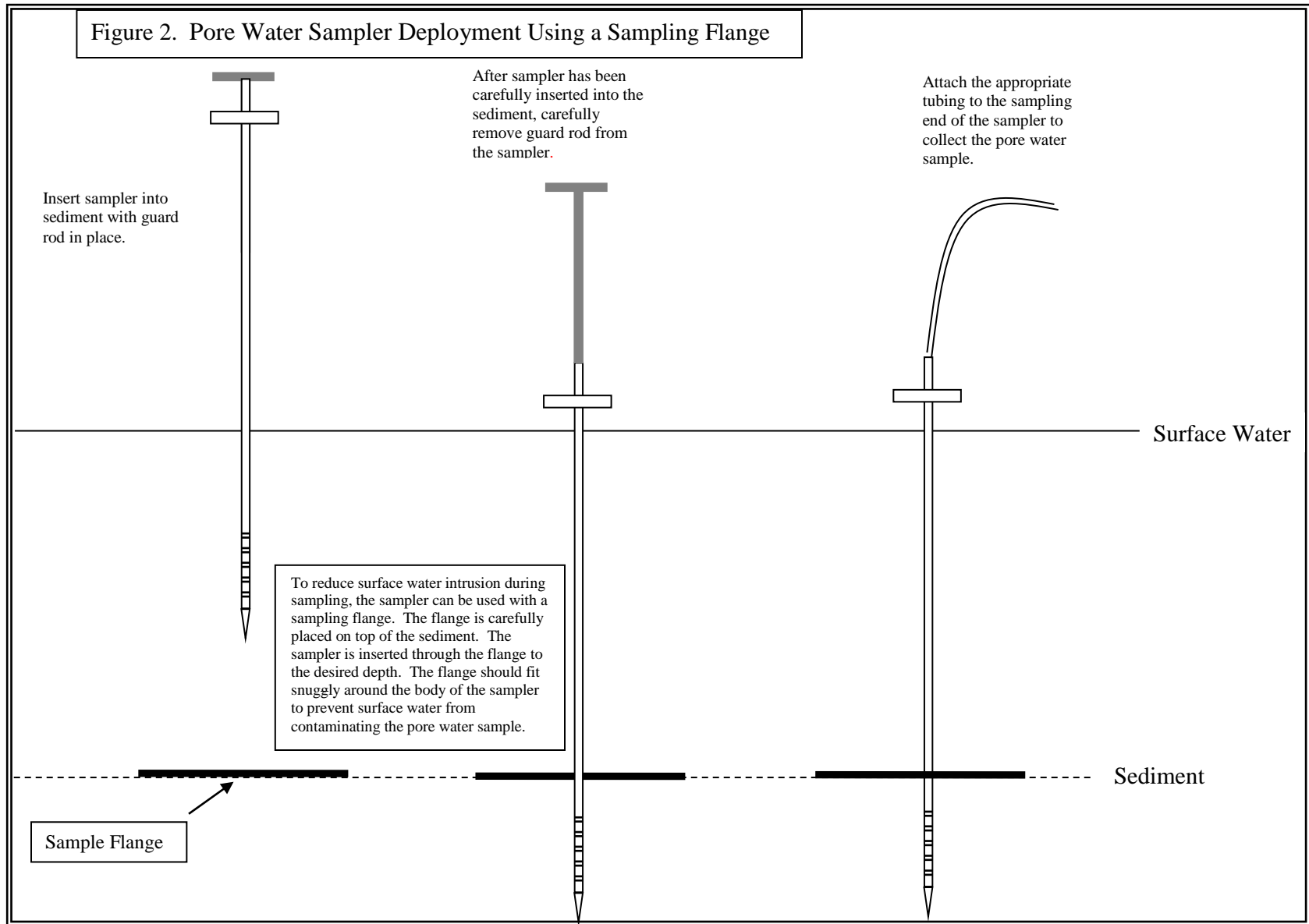


Figure 3. Pore Water Sampler Deployment Using a Peristaltic Pump to Sample

