Appendix 3

Standard Operating Procedure

for the USGS Reston, Virginia Environmental Organic Geochemistry Laboratory

Sectioning of Sediment Cores

Note: A version of this Standard Operating Procedure was accepted by the National Oceanic and Atmospheric Administration (NOAA) in August 1993 as part of the Analytical Chemistry Quality Assurance Plan for the Southern California Natural Resource Damage Assessment.

Contents

Sectioning of Sediment Cores

1. **Application:** This is a method for the sectioning of frozen sediment cores collected in sediment core liners. In this project the sediment cores were obtained by subcoring sediments collected in a box corer. The sediments in the subcores were frozen immediately after subsampling by storing them upright in a -20 °C freezer.

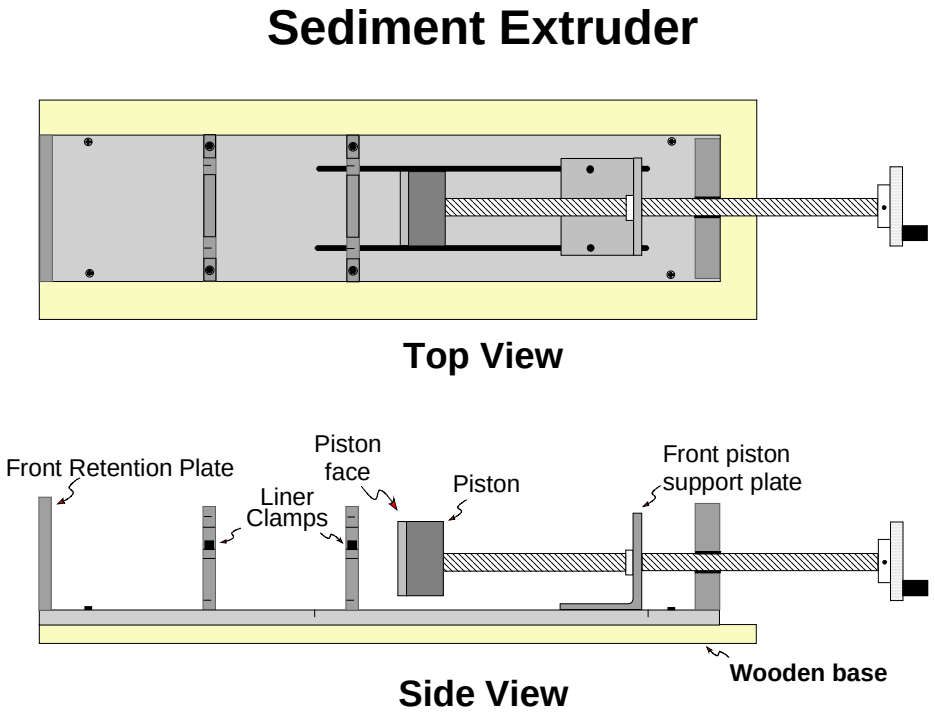
- **a.** ***Tested concentration range.*** Not applicable. For discussion of this see Appendix 4.
- **b.** ***Sensitivity.*** Not applicable. For discussion of this see Appendix 4.
- **c.** ***Detection limit.*** Not applicable. For discussion of this see Appendix 4.
- **d.** ***Interferences.*** Not applicable. For discussion of this see Appendix 4.
- **e.** ***Sectioning rate***. The procedure described here permits the sectioning of cores reaching lengths of 60 cm within a 3 hour period. Under optimal conditions, two individuals can completely section two such cores within an 8-hour day.

2. **Chemistry:** No chemical reactions are involved in this procedure.

3. **Apparatus:**

- **a.** ***Instrumentation***. No instrumentation is used in this procedure.
- **b.** ***Hardware/glassware.*** A custom-made sediment extruder is used to push discrete intervals of the frozen sediments from the core liner for sectioning. A picture of the extruder is shown in the schematic below.

Figure 1. Sediment extruder used in sediment core sectioning.



Glassware and implements used in this procedure include the following:

-8-oz. wide-mouth glass jars with Teflon™-lined caps (I-CHEM™ Certified 200 series; p/n 220-0250)
-heavy duty broad blade (≥ 3" wide) putty knife (1)
-putty knife (1¼" wide blade; 1)
-canvas rock bags (10.5 x 19 cm, 2)
-heat gun
-knife or razor blade
-stainless steel spatulas (2)
-beakers: 1000 mL
-500-mL Teflon™ squeeze bottles (2)
-heavy duty aluminum foil, solvent rinsed on shiny side
-hack saws (20 pt; 2)
-"C" clamps (3)
-Phillips head screw driver
-labels, notebook, short 10 cm metric ruler with mm divisions
-bottle brushes, sponges
-steel wool
-disposable latex gloves
-wooden or rubber mallet
-hammer

c. ***Chemicals.*** Chemicals used in this procedure include the following:

-glass-distilled methanol (Burdick & Jackson, GC^2 grade)
-glass-distilled dichloromethane (Burdick & Jackson, High Purity grade)
-dry ice
-Alcojet™ detergent

4. Standards:

a. ***Calibration standards.*** Not applicable. For discussion of this see Appendix 4.
b. ***Surrogates (recovery).*** Not applicable. For discussion of this see Appendix 4.
c. ***Internal (quantitation) standards.*** Not applicable. For discussion of this see Appendix 4.

5. Procedure:

a. ***Preparation for sectioning process.*** All materials used for core sectioning are cleaned carefully prior to removing the frozen core from the freezer. The hack saw blades, when new, are scrubbed with steel wool and washed with detergent (Alcojet™) and water, and rinsed successively with methanol and dichloromethane. After first use, the blades are not scrubbed with steel wool but are cleaned with detergent and water, followed by methanol and dichloromethane in succession. They are then placed on a solvent-rinsed piece of heavy duty aluminum. The putty knife, spatulas and the aluminum plate (on the front of the extruder piston; *cf*. Figure 1) are cleaned with tap water, methanol, and then dichloromethane. Heavy duty solvent-rinsed aluminum foil is placed where the implements are laid and also over the area where the extruder is to be

placed. Chunks of dry ice are placed inside of the rock bags and crushed using a mallet. Depending upon the length of the core, the front piston support plate (Figure 1) may need to be adjusted to allow enough room for the core to fit between the piston face and the front retention plate. This requires disassembly of the extruder from the wooden base by removal of four Phillips head wood screws. After the front piston support plate has been positioned properly, the threaded rod connected to the piston is moved back by turning the rod handle counterclockwise. The extruder is then reattached to the wooden base, and the base is clamped to the end of the bench with C clamps so that the core will be extruded toward the interior of the bench. The person sectioning the core and the assistant (who is primarily responsible for transferring sediments to the jars, keeping notes and cleaning implements) now put on disposable latex gloves.

b. ***Core sectioning***. The frozen core (in polycarbonate liner, 8 cm id x 2 mm wall thickness) is removed from the freezer and placed on a portion of the bench on which a piece of heavy duty solvent-rinsed aluminum foil has been placed. The end caps are removed from the core by cutting the electrical tape that holds them on with a knife or razor blade and gently prying them off. The subcore is then placed into the extruder with the upper sediment surface pointing toward the front retention plate and the core bottom toward the piston face. The piston is advanced into the core liner until it meets the bottom of the sediment core by rotating the rod handle clockwise. A heat gun is used to thaw the surface of the sediments inside the polycarbonate liner just enough to permit the frozen sediment core to be extruded. The sediments are then advanced about 1 cm, and the surface of the core is inspected. If an ice cap is present, the ice is gently cracked off with a stainless steel spatula onto a piece of aluminum, and the water + foil are discarded. The sediments are then extruded by turning the threaded rod clockwise driving the piston until the upper 2 cm of sediment are exposed beyond the outer edge of the front retention plate. A short ruler is used to accurately measure the distance. At this point, one or two of the rock bags filled with crushed dry ice are draped over the top of the core to keep the core frozen. It is important to cover the full length and sides of the core, especially near the piston where melting could occur.

A piece of heavy duty solvent-rinsed aluminum foil is placed in front of the extruder below the exposed sediment section. A clean hack saw is used to cut the sediment section (takes about 1-1.5 minutes) using the outer face of the front retention plate as a guide for the blade. Care must be taken in the later stages of the cutting to ensure that the sediments do not break away. The goal is to get a clean cut with the blade perpendicular to the core length at all times. Care must also be taken to "catch" the sediment section with the aluminum foil rather than letting it fall to the table where it might be damaged or roll off. Once the section has been cut, the dirty saw is placed in the sink for cleaning. With the sediment section lying flat on the aluminum foil, approximately 0.5 cm of sediment is carefully trimmed from the outside (radially) using the heavy duty broad blade putty knife and a hammer. The purpose here is to remove and isolate sediment that was in contact with the core liner and could either have been contaminated from it or been subject to smearing during the subcoring or extrusion operations.

Once the section has been cut and the outer 0.5 cm trimmed, the center portion of the secion is then broken into three or four pieces with the heavy duty broad blade putty knife using the hammer (gently rapping). The frozen sediment fragments are transferred with the small putty knife to a clean I-CHEM™ jar, previously labeled as described below. Any excess sediment that might be present on the lip of the jar is cleaned off with a disposable tissue (wiping outward) to ensure that the lid will seal properly, and the lid is screwed onto the jar as tightly as possible. The jar is then placed in the freezer (-20 °C). A running log is kept by the assistant of the intervals that are cut and the labels on the jars to ensure that no sections are mislabeled. The outer face of the front retention plate is wiped clean with a disposable tissue, the putty knives are placed in the sink for cleaning and a new piece of heavy duty solvent rinsed aluminum foil is placed below the exposed sediment section (shiny side up).

The implements are rinsed thoroughly with tap water and scrubbed with a sponge and/or brush to remove all visible traces of sediment. They are then taken to a hood where the water and any residual hydrophobic organics are removed by successive rinses of methanol and dichloromethane using Teflon™ squeeze bottles. They are placed on heavy duty solvent-rinsed aluminum foil to air dry. Meanwhile, the rock bags are removed from the core and, if necessary (usually only for the first 2-4 sections), the core liner is warmed briefly with the heat gun to defrost the sediment in contact with the inside of the liner. Another 2-cm section is extruded, and the sectioning process described above is repeated. The entire core is cut in this fashion without a break. As long as the dry ice is applied consistently there is no thawing of the core. For the longest cores (*e.g*. 60 cm) it is desirable to loosen the liner clamps (*cf*., Figure 1) and rotate the core 180° after half of the sectioning is complete so that cooling is applied more uniformly to the core. However, this is not necessary as long as the dry ice is applied consistently during sediment cutting and sectioning proceeds without delay.

c. ***Finishing touches*.** All jars with core sections are placed inside of the cardboard boxes in which they were shipped by the manufacturer and maintained in a freezer with the other sectioned cores. All implements are cleaned as described above and wrapped in solvent-rinsed aluminum foil until the next sectioning is to be performed at which time they are cleaned again to remove any traces of contamination. The extruder is removed from its wooden base, and both extruder and base are cleaned thoroughly with tap water to remove visible traces of sediment. The aluminum foil on the benches is removed, and the benches are scrupulously cleaned with water, methanol and dichloromethane to remove any contamination that may have resulted from the core-sectioning operation. The floors are swept and wet-mopped to remove residual sediment that might have fallen onto the floor.

6. **Calculations:** No calculations are required in this procedure.

7. **QA/QC Considerations:** Following are some general remarks concerning the QA/QC considerations related to the sectioning procedure. Sectioning of cores is done in order of increasing contamination starting with the cores containing the cleanest sediments. As discussed above, all implements that come in contact with the sediments are cleaned

assiduously. The sample jars purchased from I-CHEM™ are used as received without further cleaning. Two concerns are uppermost in the sectioning process: 1) to avoid cross contamination between/among sections, and 2) to avoid introduction of contamination from the laboratory environment to the sediments. The procedures implemented to avoid cross contamination include cleaning of implements between sections, cleaning of the outer face of the front retention plate, use of solvent cleaned aluminum foil for each core trimming and section cutting operation and maintaining the core in a frozen condition. In order to avoid introduction of environmental contamination, sediments are handled with freshly solvent rinsed aluminum foil and the jars are sealed tightly.

8. Health, Safety, and Waste-Disposal Information:

a. ***Personal protection*****.** Safety glasses and protective gloves are recommended whenever reagents or samples are handled. For other precautions and safety procedures, consult the Material Safety Data Sheets (MSDS) for each chemical used. They are on file in the laboratory; *http://www.ilpi.com/msds/#Manufacturers* provides links to MSDSs of most chemical companies. It is important to ensure that highly contaminated waste-impacted sediments do not contaminant the skin. If so, the affected area must be washed with soap and hot water to remove any residual material.

b. ***Electrical hazards.*** Electrical systems must conform to the National Electric Code, the National Fire Protection Association Code (NFPA 70-1971), and the American National Standards Institute (ANSI) Code (C1-1971). Consult the U.S. Geological Survey's Safety and Environmental Health Handbook (U.S. Geological Survey, 2002). Shock hazards exist inside the instruments. Only an authorized service representative or an individual with training in electronic repair should remove panels or circuit boards where voltages are greater than 20 V. The instruments require a third-wire protective grounding conductor. Three-to-two wire adapters are unsafe for these instruments.

c. ***Chemical hazards.*** Hexane, dichloromethane, and methanol are solvents used in cleaning glassware and the preparation of clean adsorbents and reagents. They also are evaluated for purity. Gloves should be worn when handling organic solvents and, whenever possible, manipulations should be conducted in a fume hood. Waste solvents accumulated during rotary evaporation or other cleaning operations should be stored in a capped glass bottle (satellite accumulation point) and arrangements made for its disposal through the USGS Materials Management Office. Likewise, excess contaminated sediment must be wrapped carefully in aluminum foil, and packaged in a suitable container for disposal through the USGS Materials Management Office.

d. ***Sharps.*** Knives, saws and razor blades should be handled with care to avoid accidental cuts. This could serve as an entry point for pathogenic microorganisms or lead to infection.

9. References:

U.S. Geological Survey. 2002. USGS Handbook 445-3-H, Safety and Environmental Health Handbook, 435p.