Appendix 4

Standard Operating Procedure

for the USGS Reston, Virginia Environmental Organic Geochemistry Laboratory

Extraction of Sediments for Determination of Trace Organics

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

Extraction of Sediments for Determination of Trace Organics

1. **Application:** This is a method for the extraction of marine sediments for isolation of trace organics such as chlorinated hydrocarbons, petroleum hydrocarbons, sterols, fatty acids (free) and other organic solvent soluble substances which will be analyzed by gas chromatography/electron capture detection and gas chromatography/mass spectrometry. In addition, this method provides for determination of the total extractable organic matter present in marine sediment samples. This is used to estimate the amount of extract necessary for instrumental analysis.

a. ***Tested concentration range***. Sediments collected from the Palos Verdes Shelf near station 3C in 1992 were analyzed by the USGS using the methodology described in this Standard Operating Procedure. Based on data from the USGS investigations (Eganhouse *et al*., 2000) and analyses performed by the LACSD (Los Angeles County Sanitation Districts; 1992, 2006), samples collected at or near LACSD stations 3C and 6C, which are under investigation here, are expected to yield concentrations of DDTs and PCBs as given in the table below:

Table 1. Estimated concentration ranges of DDT/PCB analytes.

Representative Compound	Description	Expected high concentration (µg/dry g)	Expected low concentration (µg/dry g)
p,p'-DDE	typically the most abundant DDT metabolite	180	0.01
o,p'-DDT	least abundant DDT metabolites	< 0.002[a]	< 0.0002[a]
$PCB_{5\%}$	hypothetical PCB congener representing 5% of the ΣPCB	0.949	0.011
$PCB_{.05\%}$	hypothetical PCB congener representing 0.05% of the ΣPCB	0.010	0.0001

[a]Concentration is based on estimated method detection limit (Eganhouse *et al.*, 2000).

b. ***Sensitivity.*** The sensitivities of the GC/ECD (gas chromatograph/electron capture detector) and GC/MS (gas chromatograph/mass spectrometer) instruments for chlorinated hydrocarbons and long-chain alkylbenzenes, respectively, are provided in Appendix 7, entitled, "**Instrumental Analysis for Chlorinated Hydrocarbons**" and Appendix 5 entitled, "**Instrumental Analysis for the Long-**

chain Alkylbenzenes". The sensitivity of the Cahn microbalance in the 25 mg weighing range is 0.1 μg. Accuracy of the Cahn microbalance is 0.0012% and ultimate precision is 0.1 μg.

c. ***Detection limit.*** Method Detection Limits (MDLs) for the procedures used to determine the chlorinated hydrocarbons and the long-chain alkylbenzenes are tabulated in Appendix 7 entitled, "**Instrumental Analysis for Chlorinated Hydrocarbons**" and Appendix 5 entitled, "**Instrumental Analysis for the Long-chain Alkylbenzenes**".

d. ***Interferences.*** Following is a list of potential interferences that could be encountered during the determination of the eight DDT compounds (*p,p*'- and *o,p*'-DDT, DDE, DDD + *p,p*'-DDMU and DDNU) and the 87 PCBs (including surrogates) using the DB-5 column. Generally speaking, in Palos Verdes Shelf sediments concentrations of major DDT compounds exceed those of individual PCB congeners by 2-3 orders of magnitude. Consequently, with the possible exception of *p,p*'- and *o,p*'-DDT, interferences between PCBs and DDTs are considered insignificant. On the other hand, previous work with this methodology has shown that there is an interference between one of the chlorinated hydrocarbon surrogates, PCB 198, and unknown components in NIST (National Institute of Standards and Technology) standard reference material, SRM1941a. Time permitting, we intend to explore the use of comprehensive two-dimensional gas chromatography/micro-electron capture detection (GCxGC/μECD) and GCxGC/time-of-flight mass spectrometry to overcome this interference for SRM1941a and to investigate the significance of the potential interferences indicated in Table 2. Further discussion of the potential interferences among PCB congeners and between PCB congeners and other chlorinated hydrocarbons is given in the main body of the Open-File Report.

The long-chain alkylbenzenes (LCABs) do not ordinarily suffer from interference except as described in Eganhouse *et al*. (1983), Eganhouse (1986) and Zeng *et al*. (1998). Because the LCABs are analyzed using GC/MS, these interferences can effectively be overcome through the judicious choice of quantitation ions as discussed in Appendix 5 entitled, "**Instrumental Analysis for the Long-chain Alkylbenzenes**". Zeng *et al.* (1998) conducted an investigation of the potential effects of TAB (tetrapropylene-based alkylbenzene) interference with individual LABs (linear alkylbenzenes) when ions of mass/charge = 119 and 91, respectively, were used for quantitation of the LCABs in a sediment core collected on the Palos Verdes Shelf. They found that even in this non-optimized case, the effects of coexisting TABs on total LAB concentrations were minimal and did not significantly affect sediment concentration profiles.

Table 2. Potential Interferences for Chlorinated Hydrocarbons.

Analyte	Potential Interference
p,p'-DDT	PCB 130
o,p'-DDT	PCBs 114,131,122
p,p'-DDE	PCB 85

Table 2. Potential Interferences for Chlorinated Hydrocarbons, cont'd.

o,p'-DDE	PCB 84
p,p'-DDD	PCB 134
o,p'-DDD	PCB 110,77
p,p'-DDMU	PCB 60,56
PCB 40, (103)	elemental sulfur (S_8)
PCB 130	*p,p*'-DDT
PCBs 114,131,122	*o,p*'-DDT
PCB 85	*p,p*'-DDE
PCB 84	*o,p*'-DDE
PCB 134	*p,p*'-DDD
PCB 110,77	*o,p*'-DDD
PCB 60,56	*p,p*'-DDMU

e. ***Extraction rate***. The extraction procedure described here permits the preparation of approximately 6 samples (including blanks, SRMs [standard reference materials], *etc.*) in a 32-hour working period. This is ordinarily done over a period of approximately 4 days and includes the concentration of the combined extract to a volume suitable for gravimetric analysis of total extractable organics (TEO). This estimated rate does not include preparation of glassware and reagents.

2. **Chemistry:** The only chemical reaction that occurs in the procedure described here is that between elemental sulfur and activated copper. This reaction occurs in the step prior to gravimetric analysis at which point elemental sulfur coextracted from the sediment is removed by placing granules of activated copper into the flask containing the concentrated extract. Sulfur reacts at the surface of the copper, presumably forming CuS, witnessed as a black coating on the copper granules. A variation of this procedure has been described by Blumer (1957).

3. **Apparatus:**

a. ***Instrumentation***. The only pieces of instrumentation used in this part of the procedure are the balances.

-Cahn Model 29 (or 31) electronic microbalance
-Ohaus Model GA200D top-loading balance
-Baxter Scientific Model DX-41 drying oven

b. ***Hardware/glassware.*** A Soxhlet apparatus is used for the extraction of the sediments. This consists of the following parts:

-medium Soxhlet condensers (6; Pyrex 45/50 Model 3840-MCO)

-medium Soxhlet extraction chambers (85 mL capacity, 6)
-Cellulose extraction thimbles (33 mm x 118 mm; 6)
-6-position Electrothermal Electromantle ME heating mantle (250-mL round bottom flasks)
-Recirculating cooler for condensers: Neslab Model RTE-110

Miscellaneous pieces of equipment used in this procedure include the following:

-Bransonic Model 3200 ultrasonic cleaner
-Custom-made four-position stainless steel nitrogen gas blowdown system
-Lindberg/Blue M Model SW-11TA-1 drying oven
-Büchi Model R200 rotary evaporator with V-800 vacuum controller
-Thermolyne/Sybron Type 30400 furnace

Glassware and implements used in this procedure include the following:

-250-mL flat bottom flasks with 24/40 taper ground glass joint
-500-mL flat bottom flasks with 24/40 taper ground glass joint
-glass rod, 6 mm
-stainless steel spatula
-beakers: 200 mL
-Chemware Teflon™ boiling stones (previously extracted)
- Teflon™ squirt bottles: 500 mL
-8" stainless steel forceps
-24/40 ground glass stoppers
-aluminum pans for determining water content
-½ dram, 1-dram borosilicate vials (National Scientific #B7800-1, B7800-2)
-microsyringes: 10, 25, 50, 100, 250, 500 and 1000 µL
-separatory funnels: 500 mL
-graduated cylinders: 50, 100, 250 and 500 mL
-all glass B-D syringes: 5 mL
-blunt tip 18 gauge x 6" Popper pipetting needles
-reagent bottles: 250 mL, 500 mL
-aluminum weighing pans (for Cahn microbalance)

c. ***Chemicals.*** For preparation/testing of chemicals listed here see Appendix 1. Chemicals used in this procedure include the following:

-glass-distilled methanol (Burdick & Jackson, High Purity grade)
-glass-distilled dichloromethane (Burdick & Jackson, High Purity grade)
-glass-distilled hexane (Burdick & Jackson, High Purity grade)
-anhydrous sodium sulfate (Mallinckrodt AR)
-sodium chloride (table salt)
-solvent extracted water for blanks
-copper granules, activated (Mallinckrodt AR)
-nitrogen gas (Valley National Gases, grade 5.0)

4. Standards:

a. ***Calibration standards.*** The calibration standards used for determination of DDT metabolites, PCBs and the long-chain alkylbenzenes are described in Appendix 7

entitled, "**Instrumental Analysis for Chlorinated Hydrocarbons**" and Appendix 5 entitled, "**Instrumental Analysis for the Long-chain Alkylbenzenes**".

b. ***Surrogates (recovery).*** The surrogates used for spiking the sediments for purposes of estimating recovery are used at different concentrations. The methods used for their preparation are described in Appendix 7 entitled, "**Instrumental Analysis for Chlorinated Hydrocarbons**" and Appendix 5 entitled, "**Instrumental Analysis for the Long-chain Alkylbenzenes**". The stock solutions and dilutions (in hexane) are tabulated below. The solutions and volumes used for spiking are based on the 'expected concentrations' in each sample and are determined by the analyst prior to initiating extraction.

Table 3. Surrogates used for sediment spiking.

	PCB/DDT Surrogates		
	(ng/ul)	(ng/ul)	(pg/ul)
Congener #	**PCB-RS-05-1/1**	**PCB-RS-05-1/100**	**PCB-RS-05-1/1000**
30	213.2	2.13	213.2
121	119.3	1.19	119.3
198	77.9	0.78	77.9
	Linear alkylbenzene Surrogates		
	(ng/ul)	(ng/ul)	(ng/ul)
Compound	**LAB-RS-01-1/1**	**LAB-RS-01-1/10**	**LAB-RS-01-1/100**
1-phenyldecane	405.1	40.5	4.05
1-phenylundecane	542.2	54.2	5.42
1-phenyldodecane	504.2	50.4	5.04
1-phenyltridecane	604.5	60.4	60.4
1-phenyltetradecane	404.4	40.4	4.04

c. ***Internal (quantitation) standards.*** The internal (quantitation) standards used for determination of DDTs, PCBs and the long-chain alkylbenzenes are described in Appendix 7 entitled, "**Instrumental Analysis for Chlorinated Hydrocarbons**" and Appendix 5 entitled, "**Instrumental Analysis for the Long-chain Alkylbenzenes**". These appendices also explain how the samples are taken up in the internal standard solutions.

5. Procedure:

a. ***Water content (WC) determination.*** Just prior to performing extractions, frozen sediments collected by box coring (*cf.*, Appendix 3) are allowed to warm to room temperature and are manually homogenized using a clean glass rod or stainless steel spatula. An aliquot (2-4 g) of wet sediment is then transferred to a pre-weighed solvent-rinsed aluminum pan (with the sample designation marked on the bottom of the pan using a permanent marker). The pan + wet sediment is reweighed. The weight of wet sediment ($Wt_{sed\text{-}wet}$) is obtained as the difference between the weight of the pan alone and the weight of the pan + wet sediment. The remainder of the sediments in the sample jar are replaced in the freezer (-20 °C). The aluminum weighing pan containing the wet sediments is then placed in

a drying oven at 60 °C overnight (inside a hood) after which it is removed and put into a dessicator. After at least one hour in the dessicator, the pan + dry sediments are reweighed to a constant weight. The difference between the weight of the pan alone and the weight of the pan + dry sediments is the weight of dry sediments ($Wt_{sed\text{-}dry}$). This is used along with the weight of wet sediments ($Wt_{sed\text{-}wet}$) to compute the water content (*WC*) of the sediments (*cf*., equation 1, section 6).

b. ***Soxhlet extraction procedure.*** Sediment samples are mainatined in organic-free glass I-CHEM™ jars sealed with Teflon™-lined lids (*cf*., Appendix 3) stored in the freezer at -20 °C. The frozen sediments are allowed to warm to ambient temperatures, manually rehomogized, and an aliquot of wet sediment (3.5-30 g) is transferred to a pre-cleaned cellulose Soxhlet thimble stabilized in a clean glass beaker (cleaning procedures for the thimble are given in Appendix 1), the thimble + beaker having previously been tared on a top loading balance. The amount of wet sediment that is weighed into the thimble is based on the expected concentrations of the analytes. The thimble + beaker + wet sediment is then reweighed to determine the wet sample weight by difference ($Wt_{samp\text{-}wet}$).

The thimble + wet sediment is placed inside a clean Soxhlet extractor, and the sample is amended with small volumes (100-500 µL) of surrogate solutions corresponding to expected concentrations of the PCBs, DDTs and long-chain alkylbenzenes (*cf*. Table 3 above) using microsyringes. The surrogate solutions and the volumes that are used for any given sediment sample are based on analyte concentrations determined by the Los Angeles County Sanitation Districts and the USGS and the dry mass of sediments introduced to the thimble ($Wt_{samp\text{-}dry}$). The latter is computed from the water content and the wet weight of the sediment sample (*cf*., equation 2, section 6).

Twenty-five mL of glass-distilled methanol are used to rinse the beaker which was used to stabilize the thimble during the weighing procedure. This is then poured into the cellulose thimble inside the Soxhlet extractor. The Soxhlet apparatus is closed, a 250-mL boiling flask containing 175 mL of glass-distilled methanol (and a few precleaned Teflon™ boiling chips) is attached, and the condensers are cooled to 10 °C (using the recirculating cooler with a 50/50 mixture of water and antifreeze).

The sediments are extracted overnight (~18 hours) at a reflux rate of 3 cycles/hour (mantle setting = 5.2). The mantles are then turned off, and once the flasks have cooled, excess methanol is siphoned from the extraction chamber into the receiving flask *via* the sidearm. The methanol is transferred to a 500-mL separatory funnel for back extraction with 175 mL of pre-extracted water (*cf.* Appendix 1) and 50 mL of glass-distilled dichloromethane. A scoopula of precombusted NaCl is added to the separatory funnel to help break emulsions. The phases are vigorously agitated in the separatory funnel for a minimum of one minute. After the phases have separated, the dichloromethane (lower) layer is drained into a clean 500-mL boiling flask. This back extraction procedure is repeated twice more using 50 mL of glass-distilled dichloromethane each time

with the extracts being combined. The boiling flask is sealed with a glass stopper, and the dichloromethane back extract is stored in in the freezer. Meanwhile, a fresh boiling flask charged with 200 mL of glass-distilled dichloromethane and a few pre-cleaned Teflon™ boiling chips is attached to the Soxhlet apparatus. As before, this extraction is carried out overnight at a reflux rate of 3 cycles/hour (mantle setting = 2.4). The next day, the solvent is drained as described above and combined with the dichloromethane back extract of the methanol. The 500-mL boiling flask containing the combined extracts is stored in the freezer until rotovapping can be done.

c. ***Extract concentration and preliminary removal of sulfur and water.*** All extracts are concentrated to a small volume (~1-3 mL) by rotary evaporation at a temperature of 30 °C and a pressure of 475 torr (± 5%). Water and elemental sulfur are removed by adding excess precombusted (450 °C for ≥ 4 hours) anhydrous sodium sulfate and a small amount of activated copper granules (see preparation procedure for Cu^o in Appendix 1) to the flask, agitating the mixture, sealing the flask with a glass stopper, and allowing the extract to sit overnight in the dark. The next day, the copper granules are inspected for discoloration, and their condition is recorded in the laboratory notebook. If all of the copper is blackened, more activated copper is added and the extract is permitted to sit in the dark at room temperature overnight again. Otherwise, the extract is transferred quantitatively to a clean one-dram borosilicate vial using a glass 5-mL B-D syringe. The initial transfer is followed by three rinses of 0.5 mL each of glass-distilled dichloromethane (using a 5-mL glass B-D syringe). The volume is adjusted by evaporation under a stream of dry, organic-free, nitrogen gas using the blowdown system so that the TEO concentration falls within a range of approximately 2-40 µg/µl (total volume typically 3 mL). The target volume is based on prior measurements made on sediment cores collected from the Palos Verdes Shelf and analyzed by the USGS. [Note: Initial gravimetric analyses sometimes indicate that adjustment of the extract volume and reanalysis are required.] The final volume of extract (V_{extr}; in mL) is measured by comparing the sample meniscus with marks on a vial calibrated up to 3 mL in units of 100 µl and is recorded in a gravimetric analysis log sheet. At this point, the concentration of total extractable organics in the extract (C_{TEO}) can be measured.

d. ***Gravimetric analysis of extract.*** The Cahn microbalance is turned on the day before measurements are to be made in order to achieve stable conditions of operation. The microbalance is tared and calibrated according to manufacturer's instructions. After all air bubbles have been expelled, approximately 1 µl of dichloromethane is withdrawn into a 10-µl syringe. This volume of dichloromethane exceeds the capacity of the needle (~0.6-0.7 µl) and will be used to 'rinse' the extract from the syringe body and the needle upon explusion of the sample. The plunger is withdrawn such that the air-dichloromethane interface is visible at the 0.2-µl graduation mark. With the plunger held in this position, a small aliquot of extract (~3-4 µl) is drawn into the syringe. The analyst can use the position of the air-dichloromethane interface to judge approximately how much extract has been taken up. The exact volume of extract is determined by removing the needle from the extract solution, orienting the syringe in a

horizontal position with the needle slightly elevated, and withdrawing the syringe plunger until the air-extract interface is positioned at the 1-µl graduation mark. The total volume of the extract in the syringe is noted in the gravimetric analysis log book.

The sample is then transferred to a clean aluminum weighing pan placed on one weighing arm of a Cahn (Model 29 or 31) microbalance. This is done by positioning the needle tip just above the center of the braked aluminum pan, slowly and continuously depressing the syringe plunger to expel the extract (followed by the dichloromethane 'rinse'), and touching the last drop of liquid to the aluminum pan once the plunger has bottomed out. The sample of extract is allowed to dry with the balance door open for three minutes, after which the door is closed, the pan brake is released, and the mass of the residue (in µg) is measured and recorded in the log book.

The concentration of TEO in the extract (C_{TEO}; in µg/µL = mg/mL) is calculated by dividing the measured mass of the residue (in µg) by the volume of extract transferred to the aluminum pan with the syringe (in µL). Replicate measurements (at least three) are made on each sample extract such that the coefficient of variation for the replicate weighings is less than 15%. The mean concentration of TEO in the extract ($\overline{C}_{TEO}$) is calculated by averaging the replicate concentrations. The mass of total extractable organics (in mg) is computed as the product of the mean TEO concentration ($\overline{C}_{TEO}$; in mg/mL) and the total volume of extract (V_{extr}; in mL). The concentration of total extractable organics in the sediment sample (TEO_{sed}; in mg/dry g) is calculated by dividing the mass of total extractable organics by the sediment sample weight ($Wt_{samp\text{-}dry}$; in dry g) as described in section 6 (equation 3).

If the total mass of extractable organics in the sample is less than 25 mg, 100% of the extract is used for fractionation as described in Appendix 2 ("**Fractionation of Sediment and Porewater Extracts for Determination of Trace Organics"**). Otherwise, an aliquot corresponding to ≈ 25 mg is transferred to a separate ½-dram vial sealed with tha Teflon™-lined cap using a microsyringe. In either case, the aliquot of extract to be fractionated is gently evaporated until just dry under a stream of dry nitrogen gas and immediately taken up in 250 µl of hexane dried over anhydrous Na_2SO_4 (*cf*., Appendix 1). The aliquot of extract is stored in the freezer until adsorption chromatographic fractionation can be performed.

6. **<u>Calculations</u>:** The only calculations involved with this portion of the methodology are: 1) the determination of water content of the sediments (*WC*; equation 1), 2) the determination of the mass of dry sediments used in the Soxhlet extraction ($Wt_{samp\text{-}dr}$y; equation 2), and 3) the determination of the concentration of total extractable organic matter in the sediments (TEO_{sed}; equation 3).

The equation used for computing the water content of sediment samples is given below.

$$WC = \left[\frac{\left(Wt_{sed-wet} - Wt_{sed-dry} \right)}{Wt_{sed-wet}} \right] \times 100 \qquad (1)$$

where: WC = water content (%),

$Wt_{sed\text{-}wet}$ = mass of sediment sample before drying (wet g),

$Wt_{sed\text{-}dry}$ = mass of sediment sample after drying (dry g).

The equation for computing the weight of dry sediments used in the Soxhlet extraction is given below.

$$Wt_{samp-dry} = \left[1 - \frac{WC}{100} \right] \times Wt_{samp-wet} \qquad (2)$$

where: $Wt_{samp\text{-}dry}$ = mass of sediment used in Soxhlet extraction (dry g),

$Wt_{samp\text{-}wet}$ = mass of sediment used in Soxhlet extraction (wet g).

The equation for computing the concentration of total extractable organic matter in the sediment sample is given below.

$$TEO_{sed} = \frac{\overline{C}_{TEO} V_{extr}}{Wt_{samp-dry}} \qquad (3)$$

where: TEO_{sed} = concentration of total extractable organic matter in the sediment sample (mg/dry g),

$\overline{C}_{TEO}$ = mean concentration of TEO in final extract (mg/mL),

V_{extr} = total volume of final extract (mL),

7. **<u>QA/QC Considerations</u>:** Following is information on the analysis of blanks, matrix spikes, SRMs and other QA/QC considerations.

a. ***Matrix spike/matrix spike duplicate (MS/MSD).*** A Matrix Spike/Matrix Spike Duplicate pair will be processed along with every 25 field samples. These consist of sediment samples that are analyzed with and without spikes. The spike solutions used for the PCBs (PCB-SS-03-1/2) and the DDTs (DDT-SS-02-1/1) are described in Appendix 7. The spike solution for long-chain alkylbenzenes (LAB-SS-04-1/1) is described in Appendix 5.

b. ***Certified reference material/laboratory control material***. A Certified Reference Material/Laboratory Control Material consisting of NIST SRM 1941a will be

processed with every 15 field samples (~2.5 dry g SRM 1941a/analysis). These materials are maintained in the freezer (-15 to -20 °C) until use.

c. ***Blanks.*** A "blank" consisting of sand (precombusted @ 450 °C for ≥ 4 hours) rehydrated to 50% water content using freshly prepared solvent-extracted Milli-Q water (*cf.*, Appendix 1) will be processed along with every 10 field samples.

d. ***Extract storage/archiving.*** Sample extracts are stored in borosilicate vials that are sealed with Teflon™-lined caps secured by Teflon™ tape. The vials are placed in Wheaton Vial Files™ that are kept in the freezer (-15 to -20 °C). When the extract solutions are adjusted to the final extract volumes (V_{extr}) and after aliquots of extract are removed, a pencil mark is made on the label to indicate the height of the meniscus. Records are also kept in the analyst's laboratory notebook of the final extract volumes (V_{extr}) and the aliquots of extract that are removed. Together, this information forms a basis for assessing the correct solution volume at a later time. In general, evaporation of solvent from the vials is very slow at these temperatures and insignificant within the timeframe of the analyses. However, when archiving samples, it is prudent to inspect extract liquid levels at least annually to ensure that excessive losses have not occurred. Occasionally this happens due to a flawed vial mouth or broken cap. If significant evaporative losses have been found to occur, fresh dichloromethane should be added until the volume is at the last marked level and the date of this addition marked in the analyst's laboratory notebook.

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.

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 used in the extraction of samples. 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.

d. ***Gas cylinder handling*****.** Compressed gas cylinders must be handled and stored according to the Safety and Environmental Health Handbook (U.S. Geological survey, 2002). Each cylinder must be 1) carefully inspected when received, 2) securely fastened at all times with an approved chain assembly or belt, 3) capped at all times when not in use, 4) capped when transported, 5) transported only by a properly designed vehicle (hand truck), and 6) stored separately with other full, empty, flammable, or oxidizing tanks of gas, as appropriate.

e. ***Sharps.*** Microsyringes with fixed or removable needles should be handled with care to avoid accidental skin punctures.

9. References: Following are some additional sources of information about the procedures that have been described here.

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