Appendix 2

Standard Operating Procedure

for the USGS Reston, Virginia Environmental Organic Geochemistry Laboratory

Fractionation of Sediment Extracts 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

Fractionation of Sediment Extracts for Determination of Trace Organics

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1. **Application:** This is a method for the fractionation of extracts isolated from marine sediments. The procedure describes the isolation of three fractions corresponding to the saturated hydrocarbons (F1), aromatic hydrocarbons and halocarbons (including long-chain alkylbenzenes, PCBs, and DDTs; F2), and polar species including the ketones, aldehydes, and alcohols (F3) by adsorption chromatography. Only the F2 fraction is used for further analyses as part of the **Palos Verdes Remediation Project**.

a. ***Tested concentration range*.** This procedure is one part of a complete analytical method. Expected concentrations of the PCBs and DDTs in sediments under investigation as part of the **Palos Verdes Remediation Project** are given in Appendix 4. Expected concentrations of the long-chain alkylbenzenes (LCABs) in sediments can be found in Appendix 5.

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, can be found in Appendix 7, entitled, "**Instrumental Analysis for Chlorinated Hydrocarbons**" and Appendix 5 entitled, "**Instrumental Analysis for the Long-chain Alkylbenzenes**".

c. ***Detection limit.*** Method Detection Limits 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.*** A list of interferences that could possibly be encountered during the determination of the eight DDT compounds and the 87 PCBs is provided in Appendix 4. Because they are determined by GC/MS, the long-chain alkylbenzenes do not ordinarily suffer from interference except as described in Eganhouse *et al*. (1983), Eganhouse (1986), and Zeng *et al*. (1998). Using GC/MS, these interferences can effectively be overcome through the judicious choice of quantitation ions as described in Appendix 5 entitled, "**Instrumental Analysis for the Long-chain Alkylbenzenes**"**.**

e. ***Fractionation rate*.** Approximately four samples can be fractionated in a single 8-hour day. Preparation of four F2 fractions for instrumental analysis (*i.e*. fraction concentration) requires approximately four hours. Extracts can be fractionated and prepared for instrumental analysis at a rate of 1 per 3 hours.

2. **Chemistry:** This method relies upon adsorption chromatography. There are no chemical reactions involved. Rather, differences in the interactions between the solutes and the stationary and mobile phases effect the separation of analytes from interfering substances.

3. Apparatus:

a. ***Instrumentation.*** The only instrumentation required in this procedure is a high resolution gas chromatograph equipped with a hydrogen flame ionization detector (FID) and a split/splitless injector. This instrument is used for analysis of fractions collected during initial calibration of the adsorption chromatography column when a new batch of adsorbents is prepared. In our laboratory we use a Hewlett-Packard 5890 Series II gas chromatograph equipped with flame ionization detector and a split/splitless injector (including a Merlin microseal septum). The instrument is also equipped with a Hewlett-Packard 7673 autosampler. Both autosampler and gas chromatograph are controlled by a Perkin Elmer Nelson Series 900 Link interface with data acquisition and reprocessing through the PerkinElmer TotalChrom Workstation (version 6.2.1) software which operates on a Dell Optiplex GX300 computer. General instrument parameters and conditions used in the analysis of column calibration fractions are indicated in Table 1.

Table 1. Chromatographic conditions used to evaluate column calibration fractions.

Parameter	Setting
Column:	J&W Scientific
Phase	DB-5
Length	30 meters
ID	0.25 mm
Film thickness	0.25 μm
Gas Chromatograph:	HP5890 Series II
Injector	split/splitless (splitless mode used)
Injector temperature	275 °C
FID temperature	300 °C
Initial oven temperature	40 °C, 5 min isothermal hold
Program rate	6 °C/min
Final temperature	285 °C, 20 min isothermal hold
Carrier, linear velocity	helium, 30 cm/sec

b. ***Hardware/glassware***. Following is a list of the hardware and glassware needed for this procedure.

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

-Bransonic Model 3200 ultrasonic cleaner
-Custom-made stainless steel four-position nitrogen gas blowdown system
-Büchi Model R-200 rotary evaporator with a V-800 vacuum controller

-Baxter Scientific Products DX-41 gravity drying oven for adsorbent activation

Glassware and implements used in this procedure include the following:

-chromatography column: 11 mm id x 25 cm long (Kontes #420280-0213)
-glass wool, combusted at 450 °C for 4 hours
-miscellaneous beakers (50-1000 mL)
-glass B-D syringes: 5, 10 mL
-blunt tip 18-gauge, 6" Popper pipetting needles
-50-mL pear-shaped flasks with 14/20 ground glass stoppers
-cork rings for pear-shaped flasks
-microsyringes: 250, 500, and 1000 µL
-10-cc glass syringe pipettes
-Teflon™ rod
-½-, 1-dram borosilicate vials (National Scientific #B7800-1, B7800-2)
-Target™ amber DP vials (National Scientific, #C4000-2W)
-Target™ 250-µL conical glass inserts (National Scientific, #C4010-629L)
-500-mL Erlenmeyer flasks with ground glass joints and glass stoppers
-marking pen
-stainless steel forceps
-aluminum foil

c. ***Chemicals.*** 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)
-deionized water (Milli-Q)
-sodium sulfate (Mallinckrodt AR)
-silica gel (EM Science, #7752-3)
-alumina (Fisher, #A941, 80-200 mesh)
-copper granules (Mallinckrodt AR)
-nitrogen gas (Valley National Gases, grade 5.0)

4. **Standards:** Column calibration is performed for each new batch of adsorbents as described below. This is the only use of standards in this procedure.

a. ***Column calibration standards.*** With each new batch of adsorbents (silica gel and alumina) that are prepared, an adsorbent calibration experiment is carried out using the procedure described in section 5.b. This is done to ensure that the targeted analytes elute within the expected chromatographic windows. Earlier studies (Eganhouse *et al*., 1989; Hendricks and Eganhouse, 1992) form the basis for this calibration procedure. Compounds corresponding to the saturated hydrocarbons (*n*-C_{22}), linear alkylbenzenes (1-phenyl-C_{14}), PCBs (congeners 4, 69, 198), DDTs (*p,p*'-DDD), polycyclic aromatic hydrocarbons (chrysene), fatty acid methyl esters (*n*-C_{23} FAME) and alcohols (cholesterol) are used in a solution made to a concentration of ~30 ng/µL hexane/component (Table 2). These compounds were selected on the basis of their elution characteristics (Eganhouse *et al*., 1987). For example, *n*-C_{22} and 1-phenyl-C_{14} are used to define the F1/F2 cut (*n*-C_{22} falling in the F1 fraction, 1-phenyl-C_{14} falling in the F2 fraction).

Similarly, *p,p*'-DDD (the latest eluting chlorinated hydrocarbon of the analytes being measured) and *n*-C_{23} FAME (intended to represent the biogenic lipid fraction) are used to establish the F2/F3 fraction cut. Thus, *p,p*'-DDD should be recovered in the F2 fraction and *n*-C_{23} FAME in the F3 fraction.

Table 2. Solutions used for column chromatography adsorbent calibration.

		Concentration (ng/μL)		
Compound[a]	**Description**	**CCM-CS-01-1/1**	**CCM-SS-01-1/2**	**CCM-IS-01-1/2**
PCB 4	analyte	28.78	28.78	
PCB 69	analyte	27.36	27.36	
1-phenyl-C_{14}	analyte	37.43	37.43	
***n*-C_{22}**	analyte	37.19	37.19	
***p,p*'-DDD**	analyte	35.93	35.93	
Chrysene	analyte	20.00	20.00	
PCB 198	analyte	30.04	30.04	
***n*-C_{23} FAME**	analyte	33.19	33.19	
Cholestane	internal std	29.98		29.98
Cholesterol	analyte	27.74	27.74	

[a]In approximate order of elution. Note: CS=calibration standard solution, SS=spike solution, IS=internal standard solution.

5. Procedure:

a. ***Preparation of adsorbents.*** Approximately 200 g of 80-200 mesh silica gel (EM Science, EM-7752-3) and 100 g of 80-200 mesh basic alumina (Fisher A941) are each weighed into individual one-liter beakers to which 500 mL of redistilled methanol is added. The mixture is sonicated for 30 minutes after which the methanol is decanted. The adsorbents are rinsed three times with 50-100 mL of dichloromethane (DCM) with decanting, and the adsorbents are then sonicated with 500 mL DCM for 30 minutes. The DCM is decanted, and the beaker is covered with aluminum foil with needle punctures to permit escape of vapors but prevent entry of macroscopic particulate matter. The adsorbents are allowed to dry in the hood overnight or until dry. Once dry, the adsorbents are transferred to pre-tared, glass-stoppered 500-mL Erlenmeyer flasks, and the silica gel is activated at 180 °C overnight. Alumina is activated overnight at 250 °C for 18 hours. The flasks are removed from the oven, sealed with ground glass stoppers and allowed to come to room temperature. The flasks are then reweighed to determine the mass of activated adsorbent. Previously tested, dichloromethane-extracted, redistilled Milli-Q water (see Appendix 1) is added to each flask in sufficient volumes to equal 3% (w/w) of the activated adsorbents. This is ordinarily accomplished by weighing a 5-mL B-D syringe loaded with the targeted volume of water prior to and after the transfer has been made. The weight difference divided by the mass of adsorbent (times 100) gives the % deactivation. The flasks are shaken vigorously until all clumps are broken up (a couple of minutes) and are allowed to sit overnight in the dark (stoppered) to equilibrate. The next day the adsorbents are covered with redistilled, dry hexane (maintained in the dark over anhydrous sodium sulfate) and are stored in a cabinet

unless adsorbents are needed for column chromatography. Generally, the adsorbents are used within a few days of preparation. Prolonged storage (*e.g.* >1 week), particularly under humid conditions, can lead to further deactivation, invalidating the calibration. This is to be avoided.

b. ***Calibration of the chromatography column.*** The procedure for preparation of the adsorbents is given in section 5.a. The procedure for column packing is given in section 5.c. For purposes of the initial adsorption chromatography column calibration, a 250-µL aliquot of a column calibration spike solution (*e.g.* CCM-SS-01-1/2; Table 2) is introduced to a column. Fractions are collected according to the schedule shown in Table 3. Comparison of the fraction collection schedule given in Table 3 with the chromatographic separation procedure described in section 5.c. for sample extracts reveals that the initial calibration test involves collection of more sub-fractions at the margins of the F2 fraction. This is to help assess the elution of the calibration compounds in the vicinity of the F1/F2 and F2/F3 cut points. Each of the fractions is concentrated by rotary evaporation as described in section 5.d., transferred to separate ½-dram autosampler vials, gently evaporated to dryness and taken up in 250 µL of 5α(H)-cholestane (~30 ng/µL; *e.g*. CCM-IS-01-1/2; Table 2). A 1-µL aliquot of each fraction is then analyzed by GC/FID (gas chromatography/flame ionization detection) using the conditions given in Table 1. Quantitation is by the internal standard method using a separate solution containing 5α(H)-cholestane + the column calibration analytes at a concentration of ~30 ng/µL/component for purposes of GC/FID single-point calibration (*e.g*. CCM-CS-01-1/1; Table 2). Use of a given batch of adsorbents for sample processing depends upon the success of the initial calibration experiment, criteria for which are given in section 7 of this document.

Table 3. Fractionation scheme for calibration of adsorption chromatography column.

Fraction	Volume/solvent
F1a	10 mL hexane
F1b	5 mL hexane
F2a	5 mL hexane
F2b	25 mL 30% DCM in hexane
F2c	5 mL 30% DCM in hexane
F3a	5 mL methanol
F3b	35 mL methanol

c. ***Chromatographic separation.*** The goal of the following procedure is to isolate a single fraction (F2) containing the long-chain alkylbenzenes, the eight targeted DDT compounds and all of the polychlorinated biphenyls from an aliquot of the total extract of a sediment sample. As described in Appendix 4, aliquots of the final extract of sediments bearing ≤ 25 mg of TEO (total extractable organics) will have been transferred to 250 µL of redistilled hexane. This aliquot is stored in the freezer in a clean ½-dram vial until adsorption chromatographic separation is to take place.

A plug of pre-combusted glass wool is introduced with clean stainless steel forceps into a 1.1 cm id x 25 cm column equipped with a Teflon™ stopcock. The inner walls of the column are rinsed with approximately 10 mL of dry redistilled hexane to remove glass fibers and any residual contaminants in the apparatus. The hexane is allowed to run through the stopcock with the waste being collected in a beaker. The stopcock is closed, and an additional 5 mL of dry hexane is added.

Silica gel (prepared as described above) is added to the column as a slurry in hexane using a clean 10-mL syringe pipette (with the tip truncated). With intermittent tapping using a Teflon™ rod (to pack the column and remove bubbles) and additions of the slurry, the column is wet packed to a height of 12.0 cm. This distance is marked with a fine tip marker pen from the top of the glass wool to the top of the silica gel. The walls of the column are rinsed with dry hexane (stored over anhydrous Na_2SO_4) to prevent the buildup of dry silica gel during packing. Also, it is important to keep the silica gel covered with solvent. The same procedure is used with a separate pipette syringe to form a 6.0-cm layer of alumina overlying the silica gel. Any particles adhering to the sides of the column during the final stages of the packing of silica gel and alumina are rinsed down with hexane. The solvent level is lowered to 1-2 cm above the alumina surface, and the stopcock is closed.

Using a clean stainless steel spatula, a 0.5-cm layer of anhydrous sodium sulfate is added to the column. This prevents disruption of the surface adsorbent layer during subsequent solvent additions and ensures that any residual water in the extract is removed. The solvent is then lowered to the surface of the sodium sulfate. The column is covered with a small clean beaker to prevent entry of particulate matter whenever manipulations are not being performed.

Prior to introduction of the sample, 10 mL of dry hexane is run through the column as a pre-rinse. The solvent is lowered to the alumina surface. The sample aliquot is then transferred from a ½-dram vial to the column using a 500-µl microsyringe. Care is taken to gently deliver the sample to the column head by allowing the needle to rest against the inside of the column wall while slowly depressing the plunger. After the transfer is complete, the liquid level is lowered to the alumina surface. Three successive volumes of dry hexane (150 µl each) are used to rinse the vial and the column walls (*i.e.* use hexane to rinse the vial, and then remove this to rinse the column walls). The solvent level is again lowered to the alumina surface.

A clean 50-mL pear-shaped flask is placed beneath the column (a cork ring is used for stability) for collection of the F1 fraction (saturated hydrocarbons). Three successive 5-mL additions of hexane are made to the column using a 10-mL B-D syringe equipped with an 18-gauge needle with the column flow at a rate of ~2 mL min^{-1} (stopcock full open) until 15 mL of eluate has been collected. Flow is stopped by closing the stopcock when the last of the hexane is just at, not below, the surface of the adsorbents. (It is important to never allow the adsorbents to be exposed to air as they will dry out.). The volumes are critical, so

it is important to measure the solvent without headspace. This is the F1 fraction. The F1 fraction is not being analyzed for the **Palos Verdes Remediation Project**; it is archived for possible future use. The flask is labeled, sealed with a glass stopper, and placed in the freezer until further processing is possible.

Another 50-mL pear-shaped flask is placed under the tip of the column for collection of the F2 fraction (aromatic hydrocarbons and halocarbons including the long-chain alkylbenzenes, PCBs, and DDTs). An additional 5 mL of dry hexane is added to the column, and the stopcock is opened. The stopcock is closed when the liquid level reaches the surface of the alumina. Then 30 mL of 30% DCM in dry hexane (over anhydrous Na_2SO_4) is run through the column with continuous flow at a rate of ~2 mL min^{-1} (stopcock full open), the combined eluate (*i.e*. 5 mL hexane + 30 mL 30% DCM in hexane) being collected in the 50-mL pear-shaped flask. This is the F2 fraction which will be analyzed for DDTs, LCABs (long-chain alkylbenzenes), and PCBs as part of the **Palos Verdes Remediation Project**. The flask containing the F2 fraction is sealed with a glass stopper and placed in a cabinet using a cork ring for support.

Another 50-mL pear-shaped flask is placed under the tip of the column for collection of the F3 fraction (ketones, aldehydes, alcohols, *etc*.). Forty mL of redistilled methanol is added to the column with a new 10-mL B-D syringe. This is allowed to run through the column at a rate of ~2 mL min^{-1} until the solvent level reaches the surface of the adsorbent bed. This is the F3 fraction. The F3 fraction is not being analyzed for the **Palos Verdes Remediation Project**; it is archived for possible future use. The flask is labeled, sealed with a glass stopper, and placed in the freezer until further processing is possible.

d. ***Concentration of the F2 fraction.*** The flask containing the F2 fraction is attached to the rotary evaporator, and the F2 fraction is concentrated (at temp = 30 °C and pressure = 475 torr [± 5%]) to about 1 mL. A few grains of activated copper (*cf*., Appendix 1) are added to the flask for removal of any residual elemental sulfur; the contents are gently agitated and the flask is allowed to sit (stoppered) in the dark overnight. The next day the sample is inspected for discoloration of the copper. If the copper surface is blackened, additional copper is added and the extract is allowed to sit overnight again. Otherwise, the sample is transferred to a ½-dram vial using a 1000-µl microsyringe using the following procedure.

The F2 fraction is first withdrawn into the syringe, and, without removing the syringe needle from the interior of the 50-mL pear-shaped flask, the sample is expelled onto the inner walls of the flask. This procedure is repeated three times to dissolve any residual material that may have deposited on the flask walls during rotary evaporation. The sample is then transferred to a ½-dram vial using the same 1000-µl microsyringe after which it is gently evaporated to a volume of approximately 200-300 µL under nitrogen gas on the blowdown system. Using a separate 5-mL B-D syringe, ~0.5-0.75 mL of dichloromethane is added to the pear-shaped flask, the walls being rinsed during the addition. This rinse is transferred with the 1000-µl microsyringe and added to the ½-dram vial, after

which it is again gently evaporated to a volume of approximately 200-300 µL under nitrogen gas on the blowdown system. The rinsing/blowdown procedure is repeated with fresh dichloromethane one more time. The volume of the fraction in the ½-dram vial is now reduced to about 500 µL by gentle evaporation under nitrogen gas on the blowdown system. The ½-dram vial is then capped and stored in the freezer (≤ 15 °C) until F2 fraction preparation and analysis can be performed (*cf*., Appendices 5, 7).

6. **Calculations:** No calculations are required in this procedure save for the measurement of the concentrations of calibration standard compounds by GC/FID and computation of recoveries of these compounds during the adsorption column calibration tests. The former is accomplished by the PerkinElmer TotalChrom software as described in Appendix 7, whereas the equation for calculating recovery of the calibration standard compounds in each fraction is given in equation 1. The computed recoveries are used to determine whether the calibration standard compounds targeted for elution in the F2 fraction chromatographic window (Table 3), namely, 1-phenyl-C_{14}, PCB congeners 4, 69, 198, and *p,p*'-DDD, elute within that window or not (see section 7. below).

The equation used for computing the recoveries of the analytes in adsorption chromatography fractions is given below.

$$R_i = \left[\frac{C'_{CCi}}{C_{CCi}} \right] \times 100 \qquad (1)$$

where: R_i = recovery of calibration compound *i* in fraction (percent),
C'_{CCi} = concentration of calibration compound *i* measured in the fraction (ng/µl),
C_{CCi} = concentration of calibration compound *i* added to the column (ng/µl).

7. **QA/QC Considerations:** The types and numbers of blanks, standard reference materials (SRMs), matrix spike/matrix spike duplicates (MS/MSDs) to be processed along with samples are described in Appendix 4. The procedures that are used to clean reagents (copper, water, *etc*.) and glassware are discussed in Appendix 1. In the event that an initial column calibration test reveals that the F2 fraction analytes are eluting later or earlier than the target elution volumes given in Table 3, a decision must be made to: 1) either prepare fresh adsorbents (and conduct a separate calibration exercise), or 2) repeat the calibration test with the originally prepared adsorbents. A second calibration experiment with the originally prepared adsorbents (*i.e*. the latter case) must include more sub-fractions from the F1 and/or F3 volumes than are shown in Table 3 to determine more precisely where the cuts would need to be made for actual samples. In the event that no improvement is seen with the second calibration experiment, either the elution scheme must be modified to accurately capture the F2 fraction or a new batch of adsorbents must be prepared and a calibration test performed on the new batch. Use of adsorbents for fractionation of sediment extracts cannot proceed until successful calibration is achieved.

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, the preparation of clean adsorbents and reagents, and the fractionation of samples and adsorbent calibration solutions. 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:

References to publications cited in this document and additional sources of information about these procedures (in bold) are given below.

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