

METHOD 3630C

SILICA GEL CLEANUP

1.0 SCOPE AND APPLICATION

1.1 Silica gel (silicic acid) is a regenerative adsorbent of silica with weakly acidic properties. It is produced from sodium silicate and sulfuric acid. Silica gel can be used in column chromatography for the separation of analytes from interfering compounds of a different chemical polarity. It may be used activated, after heating to 150 - 160°C, or deactivated with up to 10% water.

1.2 This method includes guidance for standard column cleanup of sample extracts containing polynuclear aromatic hydrocarbons, derivatized phenolic compounds, organochlorine pesticides, and PCBs as Aroclors.

1.3 This method also provides cleanup procedures using solid-phase extraction cartridges for pentafluorobenzyl bromide-derivatized phenols, organochlorine pesticides, and PCBs. This technique also provides the best separation of PCBs from most single component organochlorine pesticides. When only PCBs are to be measured, this method can be used in conjunction with sulfuric acid/permanganate cleanup (Method 3665).

1.4 Other analytes may be cleaned up using this method if the analyte recovery meets the criteria specified in Sec. 8.0.

1.5 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 This method provides the option of using either standard column chromatography techniques or solid-phase extraction cartridges. Generally, the standard column chromatography techniques use larger amounts of adsorbent and, therefore, have a greater cleanup capacity.

2.2 In the standard column cleanup protocol, the column is packed with the required amount of adsorbent, topped with a water adsorbent, and then loaded with the sample to be analyzed. Elution of the analytes is accomplished with a suitable solvent(s) that leaves the interfering compounds on the column. The eluate is then concentrated (if necessary).

2.3 The cartridge cleanup protocol uses solid-phase extraction cartridges packed with 1 g or 2 g of silica gel (silicic acid) adsorbent. Each cartridge is solvent washed immediately prior to use. Aliquots of sample extracts are loaded onto the cartridges, which are then eluted with suitable solvent(s). A vacuum manifold is required to obtain reproducible results. The collected fractions may be further concentrated prior to gas chromatographic analysis.

2.4 The appropriate gas chromatographic method is listed at the end of each technique. Analysis may also be performed by gas chromatography/mass spectrometry (Method 8270).

3.0 INTERFERENCES

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks. See Sec. 8 for guidance on a reagent blank check.

3.2 Phthalate ester contamination may be a problem with certain cartridges. The more inert the column and/or cartridge material (i.e., glass or polytetrafluoroethylene (PTFE)), the less problem with phthalates. Phthalates create interference problems for all method analytes, not just the phthalate esters themselves.

3.3 More extensive procedures than those outlined in this method may be necessary for reagent purification.

4.0 APPARATUS AND MATERIALS

4.1 Chromatographic column - 250 mm long x 10 mm ID; with Pyrex® glass wool at bottom and a PTFE stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex® glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

4.2 Beakers - appropriate sizes.

4.3 Vials - 2, 10, 25 mL, glass with PTFE-lined screw-caps or crimp tops.

4.4 Muffle furnace.

4.5 Reagent bottle - appropriate sizes.

4.6 Erlenmeyer flasks - 50 and 250 mL.

4.7 Vacuum manifold: VacElute Manifold SPS-24 (Analytichem International), Visiprep (Supelco, Inc.) or equivalent, consisting of glass vacuum basin, collection rack and funnel, collection vials, replaceable stainless steel delivery tips, built-in vacuum bleed valve and gauge. The system is connected to a vacuum pump or water aspirator through a vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Silica gel for chromatography columns.

5.3.1 Silica Gel for Phenols and Polynuclear Aromatic Hydrocarbons: 100/200 mesh (Davison Chemical grade 923 or equivalent). Before use, activate for at least 16 hr. at 130°C in a shallow glass tray, loosely covered with foil.

5.3.2 Silica Gel for Organochlorine pesticides/PCBs: 100/200 mesh (Davison Chemical grade 923 or equivalent). Before use, activate for at least 16 hr. at 130°C in a shallow glass tray, loosely covered with foil. Deactivate it to 3.3% with reagent water in a 500 mL glass jar. Mix the contents thoroughly and allow to equilibrate for 6 hours. Store the deactivated silica gel in a sealed glass jar inside a desiccator.

5.4 Silica cartridges: 40 µm particles, 60 A pores. The cartridges with which this method was developed consist of 6 mL serological-grade polypropylene tubes, with the 1 g of silica held between two polyethylene or stainless steel frits with 20 µm pores. 2 g silica cartridges are also used in this method, and 0.5 g cartridges are available. The compound elution patterns must be verified when cartridges other than the specified size are used.

5.5 Sodium sulfate (granular, anhydrous), Na₂SO₄. Purify by heating at 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. A method blank must be analyzed in order to demonstrate that there is no interference from the sodium sulfate.

5.6 Eluting solvents

5.6.1 Cyclohexane, C₆H₁₂ - Pesticide quality or equivalent.

5.6.2 Hexane, C₆H₁₄ - Pesticide quality or equivalent.

5.6.3 2-Propanol, (CH₃)₂CHOH - Pesticide quality or equivalent.

5.6.4 Toluene, C₆H₅CH₃ - Pesticide quality or equivalent.

5.6.5 Methylene chloride, CH₂Cl₂ - Pesticide quality or equivalent.

5.6.6 Pentane, C₅H₁₂ - Pesticide quality or equivalent.

5.6.7 Acetone, CH₃COCH₃ - Pesticide quality or equivalent.

5.6.8 Diethyl Ether, C₂H₅OC₂H₅. Pesticide quality or equivalent. Must be free of peroxides as indicated by test strips (EM Quant, or equivalent). Procedures for removal of peroxides are provided with the test strips. After cleanup, 20 mL of ethanol preservative must be added to each liter of ether.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

7.0 PROCEDURE

7.1 General Guidance

7.1.1 The procedure contains two cleanup options for the derivatized phenols and organochlorine pesticides/PCBs, but only one technique for the polynuclear aromatic hydrocarbons (PAHs) (standard column chromatography). Cleanup techniques by standard column chromatography for all analytes are found in Sec. 7.2. Cleanup techniques by solid-phase cartridges for derivatized phenols and PAHs are found in Sec. 7.3. The standard column chromatography techniques are packed with a greater amount of silica gel adsorbent and, therefore, have a greater cleanup capacity. A rule of thumb relating to cleanup capacity is that 1 g of sorbent material will remove 10 to 30 mg of total interferences. (However, capacity is also dependent on the sorbent retentiveness of the interferences.) Therefore, samples that exhibit a greater degree of sample interference should be cleaned up by the standard column technique. However, both techniques have limits on the amount of interference that can be removed. If the interference is caused by high boiling material, then Method 3640 should be used prior to this method. If the interference is caused by relatively polar compounds of the same boiling range as the analytes, then multiple column or cartridge cleanups may be required. If crystals of sulfur are noted in the extract, then Method 3660 should be utilized prior to this method. The cartridge cleanup techniques are often faster and use less solvent, however they have less cleanup capacity.

7.1.2 Allow the extract to reach room temperature if it was in cold storage. Inspect the extracts visually to ensure that there are no particulates or phase separations and that the volume is as stated in the accompanying documents. Verify that the solvent is compatible with the cleanup procedures. If crystals of sulfur are visible or if the presence of sulfur is suspected, proceed with Method 3660.

7.1.3 If the extract solvent is methylene chloride, for most cleanup techniques, it must be exchanged to hexane. (For the PAHs, exchange to cyclohexane as per Sec. 7.2.1). Follow one of the standard concentration techniques provided in each extraction method. The volume of methylene chloride should have been reduced to 1-2 mL. Add 40 mL of hexane, a fresh boiling chip and repeat the concentration as written. The final volume required for the cleanup techniques is normally 2 mL.

7.2 Standard Column Cleanup Techniques

7.2.1 Polynuclear aromatic hydrocarbons

7.2.1.1 Before the silica gel cleanup technique can be utilized, the extract solvent must be exchanged to cyclohexane. The exchange is performed by adding 4 mL of cyclohexane following reduction of the sample extract to 1-2 mL using an appropriate concentration technique (e.g., K-D using two-ball micro-snyder column) found in the 3500 series methods. The final extract volume is 2.0 mL.

CAUTION: When the volume of solvent is reduced below 1 mL, semivolatile analytes may be lost. If the extract goes to dryness, the extraction must be repeated.

7.2.1.2 Prepare a slurry of 10 g of activated silica gel (Sec. 5.3.1) in methylene chloride and place this into a 10 mm ID chromatographic column. Tap the column to

settle the silica gel and elute the methylene chloride. Add 1 to 2 cm of anhydrous sodium sulfate to the top of the silica gel.

7.2.1.3 Pre-elute the column with 40 mL of pentane. The rate for all elutions should be about 2 mL/min. Discard the eluate and, just prior to exposure of the sodium sulfate layer to the air, transfer the 2 mL cyclohexane sample extract onto the column using an additional 2 mL cyclohexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 25 mL of pentane and continue the elution of the column. Discard this pentane eluate.

7.2.1.4 Next, elute the column with 25 mL of methylene chloride/pentane (2:3)(v/v) into a flask for concentration. Concentrate the collected fraction to whatever volume is required (1-10 mL). Proceed with HPLC (Method 8310) or GC analysis (Method 8100). Validated components that elute in this fraction are:

Acenaphthene	Chrysene
Acenaphthylene	Dibenzo(a,h)anthracene
Anthracene	Fluoranthene
Benzo(a)anthracene	Fluorene
Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
Benzo(b)fluoranthene	Naphthalene
Benzo(g,h,i)perylene	Phenanthrene
Benzo(k)fluoranthene	Pyrene

7.2.2 Derivatized Phenols

7.2.2.1 This silica gel cleanup procedure is performed on sample extracts that have undergone pentafluorobenzyl bromide derivatization, as described in Method 8041. The sample extract must be in 2 mL of hexane at this point.

7.2.2.2 Place 4.0 g of activated silica gel (Sec. 5.3.1) into a 10 mm ID chromatographic column. Tap the column to settle the silica gel and add about 2 g of anhydrous sodium sulfate to the top of the silica gel.

7.2.2.3 Pre-elute the column with 6 mL of hexane. The rate for all elutions should be about 2 mL/min. Discard the eluate and, just prior to exposure of the sodium sulfate layer to the air, pipet onto the column 2 mL of the hexane solution that contains the derivatized sample or standard. Elute the column with 10.0 mL of hexane and discard the eluate.

7.2.2.4 Elute the column, in order, with 10.0 mL of 15% toluene in hexane (Fraction 1); 10.0 mL of 40% toluene in hexane (Fraction 2); 10.0 mL of 75% toluene in hexane (Fraction 3); and 10.0 mL of 15% 2-propanol in toluene (Fraction 4). All elution mixtures are prepared on a volume:volume basis. Elution patterns for the phenolic derivatives are shown in Table 1. Fractions may be combined, as desired, depending upon the specific phenols of interest or level of interferences. Proceed with GC analysis.

7.2.3 Organochlorine Pesticides and PCBs

7.2.3.1 Transfer a 3 g portion of deactivated silica gel (Sec. 5.3.2) into a 10 mm ID glass chromatographic column and top it with 2 to 3 cm of anhydrous sodium sulfate.

7.2.3.2 Add 10 mL of hexane to the top of the column to wet and rinse the sodium sulfate and silica gel. Just prior to exposure of the sodium sulfate layer to air, stop the hexane eluate flow by closing the stopcock on the chromatographic column. Discard the eluate.

7.2.3.3 Transfer the sample extract (2 mL in hexane) onto the column. Rinse the extract vial twice with 1 to 2 mL of hexane and add each rinse to the column. Elute the column with 80 mL of hexane (Fraction I) at a rate of about 5 mL/min. Remove the collection flask and set it aside for later concentration. Elute the column with 50 mL of hexane (Fraction II) and collect the eluate. Perform a third elution with 15 mL of methylene chloride (Fraction III). The elution patterns for the organochlorine pesticides, Aroclor-1016, and Aroclor-1260 are shown in Table 2.

7.2.3.4 Prior to gas chromatographic analysis, the extraction solvent must be exchanged to hexane. Fractions may be combined, as desired, depending upon the specific pesticides/PCBs of interest or level of interferences. Analyze Fraction I containing PCBs separated from most pesticides by Method 8082. Use Method 8081 to analyze for organochlorine pesticides.

7.3 Cartridge Cleanup Techniques

7.3.1 Cartridge Set-up and Conditioning

7.3.1.1 Arrange the 1 g silica cartridges (2 g for phenol cleanup) on the manifold in the closed-valve position. Other size cartridges may be used, however the data presented in the Tables are all based on 1 g cartridges for pesticides/PCBs and 2 g cartridges for phenols. Therefore, supporting recovery data must be developed for other sizes. Larger cartridges will probably require larger volumes of elution solvents.

7.3.1.2 Turn on the vacuum pump and set pump vacuum to 10 inches (254 mm) of Hg. Do not exceed the manufacturer's recommendation for manifold vacuum. Flow rates can be controlled by opening and closing cartridge valves.

7.3.1.3 Condition the cartridges by adding 4 mL of hexane to each cartridge. Slowly open the cartridge valves to allow hexane to pass through the sorbent beds to the lower frits. Allow a few drops per cartridge to pass through the manifold to remove all air bubbles. Close the valves and allow the solvent to soak the entire sorbent bed for 5 minutes. Do not turn off the vacuum.

7.3.1.4 Slowly open cartridge valves to allow the hexane to pass through the cartridges. Close the cartridge valves when there is still at least 1 mm of solvent above the sorbent bed. Do not allow cartridges to become dry. If cartridges go dry, repeat the conditioning step.

7.3.2 Derivatized Phenols

7.3.2.1 Reduce the sample extract volume to 2 mL prior to cleanup. The extract solvent must be hexane and the phenols must have undergone derivatization by pentafluorobenzyl bromide, as per the appropriate method.

7.3.2.2 Transfer the extract to the 2 g cartridge that has been conditioned as described in Sec. 7.3.1. Open the cartridge valve to allow the extract to pass through the cartridge bed at approximately 2 mL/minute.

7.3.2.3 When the entire extract has passed through the cartridges, but before the cartridge becomes dry, rinse the sample vials with an additional 0.5 mL of hexane, and add the rinse to the cartridges to complete the quantitative transfer.

7.3.2.4 Close the cartridge valve and turn off the vacuum after the solvent has passed through, ensuring that the cartridge never gets dry.

7.3.2.5 Place a 5 mL vial or volumetric flask into the sample rack corresponding to the cartridge position. Attach a solvent-rinsed stainless steel solvent guide to the manifold cover and align with the collection vial.

7.3.2.6 Add 5 mL of hexane to the cartridge. Turn on the vacuum pump and adjust the pump pressure to 10 inches (254 mm) of Hg. Allow the solvent to soak the sorbent bed for 1 minute or less. Slowly open the cartridge valve, and collect the eluate (this is Fraction 1, and should be discarded).

NOTE: If cartridges smaller than 2 g are used, then Fraction 1 cannot be discarded, since it contains some of the phenols.

7.3.2.7 Close the cartridge valve, replace the collection vial, and add 5 mL of toluene/hexane (25/75, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial. This is Fraction 2, and should be retained for analysis.

7.3.2.8 Adjust the final volume of the eluant to a known volume which will result in analyte concentrations appropriate for the project requirements (normally 1 - 10 mL) using techniques described in an appropriate 3500 series method. Table 3 shows compound recoveries for 2 g silica cartridges. The cleaned up extracts are ready for analysis by Method 8041.

7.3.3 Organochlorine Pesticides/PCBs

NOTE: The silica cartridge procedure is appropriate when polychlorinated biphenyls are known to be present.

7.3.3.1 Reduce the sample extract volume to 2 mL prior to cleanup. The extract solvent must be hexane.

7.3.3.2 Use the 1 g cartridges conditioned as described in Sec. 7.3.1.

7.3.3.3 Transfer the extract to the cartridge. Open the cartridge valve to allow the extract to pass through the cartridge bed at approximately 2 mL/minute.

7.3.3.4 When the entire extract has passed through the cartridges, but before the cartridge becomes dry, rinse the sample vials with an additional 0.5 mL of solvent, and add the rinse to the cartridges to complete the quantitative transfer.

7.3.3.5 Close the cartridge valve and turn off the vacuum after the solvent has passed through, ensuring that the cartridge never goes dry.

7.3.3.6 Place a 5 mL vial or volumetric flask into the sample rack corresponding to the cartridge position. Attach a solvent-rinsed stainless steel solvent guide to the manifold cover and align with the collection vial.

7.3.3.7 Add 5 mL of hexane to the cartridge. Turn on the vacuum pump and adjust the pump pressure to 10 inches (254 mm) of Hg. Allow the solvent to soak the sorbent bed for 1 minute or less. Slowly open the cartridge valve and collect the eluate into the collection vial (Fraction 1).

7.3.3.8 Close the cartridge valve, replace the collection vial, and add 5 mL of diethyl ether/hexane (50/50, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial (Fraction 2).

7.3.3.9 Adjust the final volume of each of the two fractions to a known volume which will result in analyte concentrations appropriate for the project requirements (normally 1 - 10 mL) using techniques described in an appropriate 3500 series method. The fractions may be combined prior to final adjustment of volume, if analyte fractionation is not required. Table 4 shows compound recoveries for 1 g silica cartridges. The cleaned up extracts are ready for analysis by Methods 8081 for organochlorine pesticides or 8082 for PCBs.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 3600 for cleanup procedures.

8.2 A reagent blank (consisting of the elution solvents) must be passed through the column or cartridge and checked for the compounds of interest, prior to the use of this method. This same performance check is required with each new lot of adsorbent or cartridges. The level of interferences must be below the method detection limit before this method is performed on actual samples.

8.3 The analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples. See the attached Tables for acceptable recovery data. For compounds that have not been tested, recovery must be $\geq 85\%$.

8.3.1 Before any samples are processed using the solid-phase extraction cartridges, the efficiency of the cartridge must be verified. A recovery check must be performed using standards of the target analytes at known concentration. Only lots of cartridges that meet the recovery criteria for the spiked compounds can be used to process the samples.

8.3.2 A check should also be performed on each individual lot of cartridges and for every 300 cartridges of a particular lot.

8.4 For sample extracts that are cleaned up using this method, the associated quality control samples should also be processed through this cleanup method.

9.0 METHOD PERFORMANCE

9.1 Table 1 provides performance information on the fractionation of phenolic derivatives using standard column chromatography.

9.2 Table 2 provides performance information on the fractionation of organochlorine pesticides and Aroclors using standard column chromatography.

9.3 Table 3 shows recoveries of derivatized phenols obtained using 2 g silica cartridges.

9.4 Table 4 shows recoveries and fractionation of organochlorine pesticides obtained using 1 g silica cartridges.

10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.
2. U.S. EPA "Evaluation of Sample Extract Cleanup Using Solid-Phase Extraction Cartridges," Project Report, December 1989.

TABLE 1
SILICA GEL FRACTIONATION OF PFBBR DERIVATIVES

Parameter	Percent Recovery by Fraction ^a			
	1	2	3	4
2-Chlorophenol		90	1	
2-Nitrophenol			9	90
Phenol		90	10	
2,4-Dimethylphenol		95	7	
2,4-Dichlorophenol		95	1	
2,4,6-Trichlorophenol	50	50		
4-Chloro-3-methylphenol		84	14	
Pentachlorophenol	75	20		
4-Nitrophenol			1	90

^a Eluant composition:

- Fraction 1 - 15% toluene in hexane.
- Fraction 2 - 40% toluene in hexane.
- Fraction 3 - 75% toluene in hexane.
- Fraction 4 - 15% 2-propanol in toluene.

Data from Reference 1 (Method 604)

TABLE 2

DISTRIBUTION AND PERCENT RECOVERIES OF ORGANOCHLORINE
PESTICIDES AND PCBs AS AROCLORS IN SILICA GEL COLUMN FRACTIONS^{a,b,c,d,e}

Compound	Fraction I		Fraction II		Fraction III		Total Recovery	
	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.
	1	2	1	2	1	2	1	2
alpha-BHC ^f					82(1.7)	74(8.0)	82(1.7)	74(8.0)
beta-BHC					107(2.1)	98(12.5)	107(2.1)	98(12.5)
gamma-BHC					91(3.6)	85(10.7)	91(3.6)	85(10.7)
delta-BHC					92(3.5)	83(10.6)	92(3.5)	83(10.6)
Heptachlor	109(4.1)	118(8.7)					109(4.1)	118(8.7)
Aldrin	97(5.6)	104(1.6)					97(5.6)	104(1.6)
Heptachlor epoxide					95(4.7)	88(10.2)	95(4.7)	88(10.2)
Technical chlordane	14(5.5)	22(5.3)	19(6.8)	39(3.6)	29(5.0)	37(5.1)	62(3.3)	98(1.9)
Endosulfan I					95(5.1)	87(10.2)	95(5.1)	87(10.2)
4,4'-DDE	86(5.4)	94(2.8)					86(5.4)	94(2.8)
Dieldrin					96(6.0)	87(10.6)	96(6.0)	87(10.6)
Endrin					85(10.5)	71(12.3)	85(10.5)	71(12.3)
Endosulfan II					97(4.4)	86(10.4)	97(4.4)	86(10.4)
4,4'-DDD ^f					102(4.6)	92(10.2)	102(4.6)	92(10.2)
Endrin aldehyde					81(1.9)	76(9.5)	81(1.9)	76(9.5)
Endosulfan sulfate					93(4.9)	82(9.2)	93(4.9)	82(9.2)
4,4'-DDT ^f			86(13.4)	73(9.1)	15(17.7)	8.7(15.0)	101(5.3)	82(23.7)
4,4'-Methoxychlor					99(9.9)	82(10.7)	99(9.9)	82(10.7)
Toxaphene ^f			15(2.4)	17(1.4)	73(9.4)	84(10.7)	88(12.0)	101(10.1)
Aroclor-1016	86(4.0)	87(6.1)					86(4.0)	87(6.1)
Aroclor-1260	91(4.1)	95(5.0)					91(4.1)	95(5.0)

TABLE 2
(Continued)

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- ^a Effluent composition: Fraction I, 80 mL hexane; Fraction II, 50 mL hexane; Fraction III, 15 mL methylene chloride.
- ^b Concentration 1 is 0.5 µg per column for BHCs, Heptachlor, Aldrin, Heptachlor epoxide, and Endosulfan I; 1.0 µg per column for Dieldrin, Endosulfan II, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Endrin, Endrin aldehyde, and Endosulfan sulfate; 5 µg per column for 4,4'-Methoxychlor and technical Chlordane; 10 µg per column for Toxaphene, Aroclor-1016, and Aroclor-1260.
- ^c For Concentration 2, the amounts spiked are 10 times as high as those for Concentration 1.
- ^d Values given represent the average recovery of three determinations; numbers in parentheses are the standard deviation; recovery cutoff point is 5 percent.
- ^e Data obtained with standards, as indicated in footnotes b and c, dissolved in 2 mL hexane.
- ^f It has been found that because of batch-to-batch variation in the silica gel material, these compounds cross over in two fractions and the amounts recovered in each fraction are difficult to reproduce.

TABLE 3

PERCENT RECOVERIES AND ELUTION PATTERNS FOR 18
PHENOLS FROM 2 g SILICA CARTRIDGES^a

Compound	Fraction 2 Average Recovery	Percent RSD
Phenol	74.1	5.2
2-Methylphenol	84.8	5.2
3-Methylphenol	86.4	4.4
4-Methylphenol	82.7	5.0
2,4-Dimethylphenol	91.8	5.6
2-Chlorophenol	88.5	5.0
2,6-Dichlorophenol	90.4	4.4
4-Chloro-3-methylphenol	94.4	7.1
2,4-Dichlorophenol	94.5	7.0
2,4,6-Trichlorophenol	97.8	6.6
2,3,6-Trichlorophenol	95.6	7.1
2,4,5-Trichlorophenol	92.3	8.2
2,3,5-Trichlorophenol	92.3	8.2
2,3,5,6-Tetrachlorophenol	97.5	5.3
2,3,4,6-Tetrachlorophenol	97.0	6.1
2,3,4-Trichlorophenol	72.3	8.7
2,3,4,5-Tetrachlorophenol	95.1	6.8
Pentachlorophenol	96.2	8.8

^a Silica cartridges (Supelco, Inc.) were used; each cartridge was conditioned with 4 mL of hexane prior to use. Each experiment was performed in duplicate at three spiking concentrations (0.05 µg, 0.2 µg, and 0.4 µg per compound per cartridge). Fraction 1 was eluted with 5 mL hexane and was discarded. Fraction 2 was eluted with 5 mL toluene/hexane (25/75, v/v).

Data from Reference 2

TABLE 4

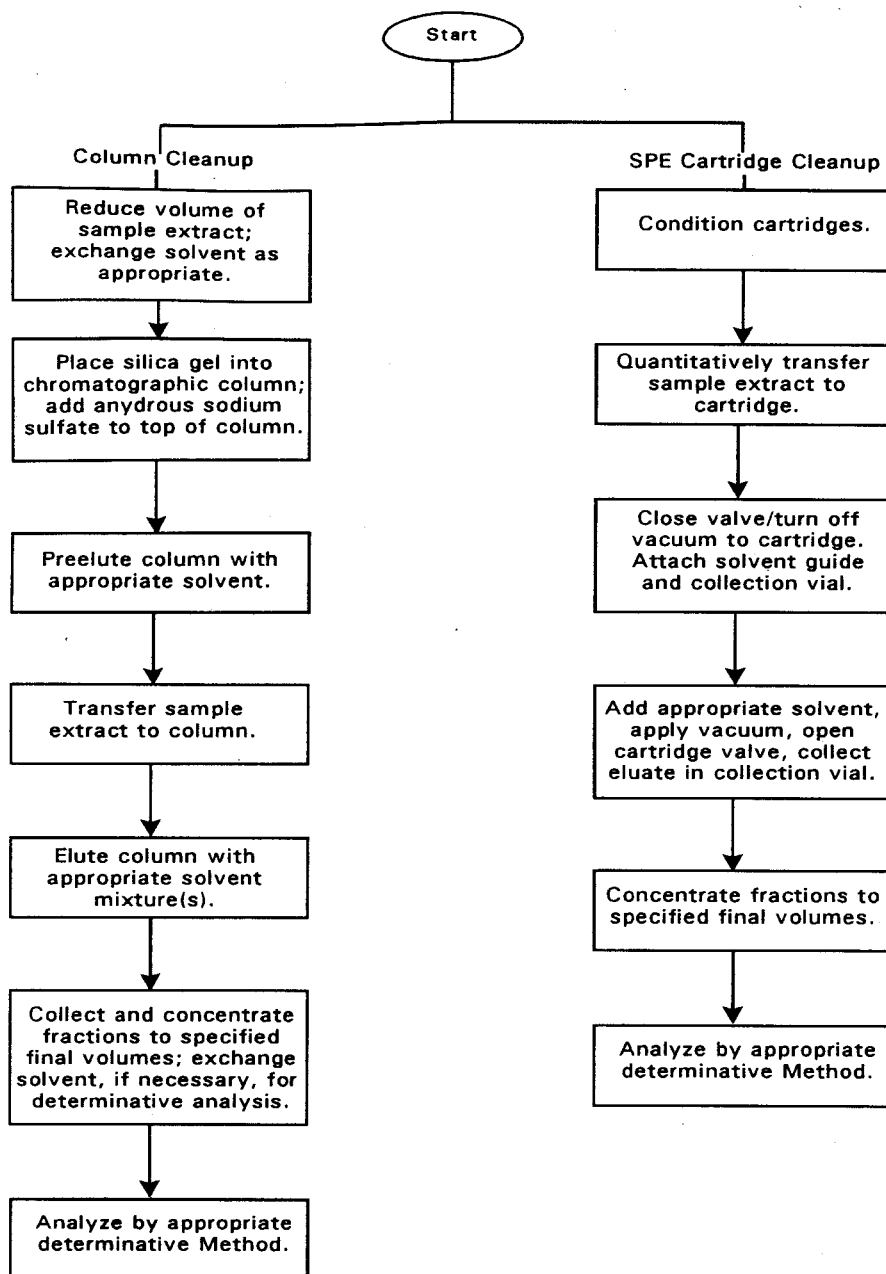
PERCENT RECOVERIES AND ELUTION PATTERNS FOR 17 ORGANOCHLORINE
PESTICIDES AND AROCLORS FROM 1 g SILICA CARTRIDGES^a

Compound	Fraction 1		Fraction 2	
	Average Recovery	Percent RSD	Average Recovery	Percent RSD
alpha-BHC	0		98.7	2.3
gamma-BHC	0		94.8	1.9
beta-BHC	0		94.3	3.0
Heptachlor	97.3	1.3	0	
delta-BHC	0		90.8	2.5
Aldrin	95.9	1.0	0	
Heptachlor epoxide	0		97.9	2.1
Endosulfan I	0		102	2.3
4,4'-DDE	99.9	1.7	0	
Dieldrin	0		92.3	2.0
Endrin	0		117	2.6
4,4'-DDD	10.7	41	92.4	3.3
Endosulfan II	0		96.0	2.2
4,4'-DDT	94.1	2.0	0	
Endrin aldehyde	0		59.7	2.6
Endosulfan sulfate	0		97.8	2.1
4,4'-Methoxychlor	0		98.0	2.4
Aroclor 1016	124			
Aroclor 1221	93.5			
Aroclor 1232	118			
Aroclor 1242	116			
Aroclor 1248	114			
Aroclor 1254	108			
Aroclor 1264	112			

^a Silica cartridges (Supelco, Inc. lot SP0161) were used; each cartridge was conditioned with 4 mL hexane prior to use. The organochlorine pesticides were tested separately from PCBs. Each organochlorine pesticides experiment was performed in duplicate, at three spiking concentrations (0.2 µg, 1.0 µg, and 2.0 µg per compound per cartridge). Fraction 1 was eluted with 5 mL of hexane, Fraction 2 with 5 mL of diethyl ether/hexane (50/50, v/v). PCBs were spiked at 10 µg per cartridge and were eluted with 3 mL of hexane. The values given for PCBs are the percent recoveries for a single determination.

Data from Reference 2

METHOD 3630C
SILICA GEL CLEANUP



Note: Select specific procedures provided in the method depending on the type(s) of analytes of interest. See the method for details regarding the appropriate elution and collection procedures.