Organochlorine Pesticides and Polychlorinated Biphenyls by Solid-Phase Extraction

UCT Part Numbers:
EEC181M6 (1000 mg C18, 6 mL Cartridge)
or
ECUNIC18 (1100mg C18, Universal Cartridge)

Compounds Recovered Using This Method

Analyte	CASRN
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Alachlor	15972-60-8
Aldrin	309-00-2
Captafol	2425-06-1
Carbophenthion	786-19-6
Chlordane - not otherwise specified (n.o.s.)	57-74-9
Chlorobenzilate	510-15-6
Chloroneb	2675-77-6
Chloropropylate	5836-10-2
Chlorothalonil	1897-45-6
Dacthal (DCPA)	1861-32-1
Diallate	2303-16-4
Dichlone	117-80-6
Dichloran	99-30-9
Dicofol	115-32-2
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Etridiazole	2593-15-9
Halowax-1000	58718-66-4
Halowax-1001	58718-67-5
Halowax-1013	12616-35-2
Halowax-1014	12616-36-3
Halowax-1051	2234-13-1
Halowax-1099	39450-05-0
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Hexachlorocyclopentadiene	77-47-4
Isodrin	465-73-6
Methoxychlor	72-43-5
Mirex	2385-85-5
Nitrofen	1836-75-5
Pentachloronitrobenzene (PCNB)	82-68-8

Permethrin (*cis* + *trans*)	52645-53-1
Perthane	72.56-0
Propachlor	1918-16-7
Strobane	8001-50-1
Toxaphene	8001-35-2
Trans-Nonachlor	39765-80-5
Trifluralin	1582-09-8
α-BHC	319-84-6
α-chlordane	5103-71-9
β-BHC	319-85-7
γ-BHC (Lindane)	58-89-9
γ-chlordane	5103-74-2
δ-BHC	319-86-8

PCBs Recovered Using This Method

Compound	CASRN	IUPAC #
Aroclor 1016	12674-11-2	-
Aroclor 1221	11104-28-2	-
Aroclor 1232	11141-16-5	-
Aroclor 1242	53469-21-9	-
Aroclor 1248	12672-29-6	-
Aroclor 1254	11097-69-1	-
Aroclor 1260	11096-82-5	-
2-Chlorobiphenyl	2051-60-7	1
2,3-Dichlorobiphenyl	16605-91-7	5
2,2',5-Trichlorobiphenyl	37680-65-2	18
2,4',5-Trichlorobiphenyl	16606-02-3	31
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	44
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	52
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	66
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	87
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	101
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	110
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	138
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	141
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	151
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	153
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	170
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	180
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	183
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	187
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	206

Procedure

1. Condition Cartridge

a) Assemble a suitable vacuum manifold system

b) Place cartridge(s) in the bulkhead fittings or cartridge adapters of the vacuum manifold

c) Attach sample transfer tube (**VMFSTFR12**) to the cartridge(s) if necessary

d) Rinse cartridge with 10 mL of methylene chloride ($MeCl_2$)

e) Let the $MeCl_2$ soak for 2 min

f) Using vacuum draw the $MeCl_2$ to waste

g) Add 10 mL of acetone. Let the acetone soak for 2 min

h) Draw the acetone to waste

i) Dry the cartridge using full vacuum for a few seconds

j) Add10 ml of methanol and allow the methanol to soak for 1 min.

Note: Do not allow the cartridge to go dry otherwise repeat starting with step 1) j)

k) Draw some of the methanol through leaving a layer just covering the frit

l) Add 20 mL of DI water. Draw most of the water through to waste but do not allow the sorbent to completely dry

2. Sample Addition

a) Adjust sample pH to ≤ 2 using 1:1 sulfuric acid

b) Mix thoroughly

c) Start vacuum and add the sample. Draw sample through the cartridge at a rate ≤ 50 mL/minute (1 L should pass through in 20 minutes or longer)

d) Allow the cartridge to dry under full vacuum for 10 min**

3. Extract Elution

a) Place a collection tube or vial under the cartridge
b) Add 5 mL of acetone to the sample bottle and swirl to remove any residue
c) Add the acetone to the cartridge. Allow the solvent to soak for 1 min then draw into collection vial
d) Repeat this procedure 3 more times using a 10 mL portions of $MeCl_2$
e) Prepare a 10-15 gram bed of anhydrous sodium sulfate anhydrous in a glass funnel using glass wool
f) Dry the extract by passing it through the funnel of sodium sulfate anhydrous
g) Carefully rinse the collection vial with $MeCl_2$, then add to the sodium sulfate, rinsing the sodium sulfate and collect

4. Concentration and Analysis

a) Carefully concentrate the extract. Solvent exchange if necessary

Note: Most extraction errors are caused by poor concentration technique

K-D Concentration Technique

Sample extracts may be concentrated to the final volume necessary by using the K-D technique or nitrogen evaporation.

a) Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to an evaporation flask
b) Collect the dried extract in the K-D concentrator
c) Rinse the collection tube and drying funnel then quantitatively transfer into the K-D flask with an additional 20 mL portion of solvent
d) Add boiling chips to the flask then attach a three-ball Snyder column
e) Attach the solvent vapor recovery glassware (condenser and collection device to the Snyder column of the K-D apparatus)
f) Pre-wet the Snyder column by adding about 1 mL of methylene chloride or acetone
g) Place the K-D apparatus on a hot water bath (15 - 20°C) above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water

h) Adjust the vertical position of the apparatus and the water temperature as necessary to complete the concentration in 10 - 20 min. At the proper rate of distillation the boiling chips of the column will actively chatter, but the chambers should not flood

i) When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 min.

j) If a solvent exchange is needed quickly remove the Snyder column, add 50 mL of the exchange solvent and a new boiling chip

k) Reattach the Snyder column. Concentrate the extract increasing the temperature of the water bath to maintain a proper distillation rate

l) Remove the Snyder column. Rinse the K-D flask and the lower joints of the Snyder column into the concentrator tube with 1 - 2 mL of solvent

m) Adjusted to a final volume of 5.0 - 10.0 mL using an appropriate solvent

Note: If further concentration is necessary, use either the micro-Snyder column technique or a N_2 evaporation technique described below

Micro-Snyder Column Technique

a) Add a fresh clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column directly to the concentrator tube

b) Attach the solvent vapor recovery glassware (condenser and collection device) to the micro-Snyder column of the K-D apparatus, following the manufacturer's instructions

c) Pre-wet the Snyder column by adding 0.5 mL of methylene chloride or the exchange solvent

d) Place the micro-concentration apparatus in a hot water bath so that the concentrator tube is partially immersed in the hot water

e) Adjust the vertical position of the apparatus and water temperature, as necessary, to complete the concentration in 5 - 10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood

f) When the apparent volume of liquid reaches 0.5 mL, remove the apparatus from the water bath and allow it to drain and cool for at least 10 min.

g) Remove the Snyder column and rinse its lower joints into the concentrator tube with 0.2 mL of solvent

h) Adjust the final extract volume to 1.0 - 2.0 mL

Nitrogen Evaporation Technique

- Place the concentrator tube in a warm bath (30° C) and evaporate the solvent to 0.5 mL using a gentle stream of clean, dry N_2 (filtered through a column of activated carbon)

Note: New plastic tubing must not be used between the carbon trap and the sample as phthalate interferences may be introduced

- Rinse down the internal wall of the concentrator tube several times with solvent during the concentration
- Position the concentrator tube to avoid condensing water into the extract
- Do not allow extract to become dry. If the volume of solvent is reduced below 1 mL, some analytes may be lost
- The extract may now be cleaned up or analyzed for analytes using the appropriate technique(s)
- If the sample is not analyzed immediately, cover the concentrator tube and store in a refrigerator. If the extract will be stored longer than 2 days, transfer to a vial with a PTFE-lined screw-cap and store in a refrigerator

**Faster drying results can be obtained by removing the cartridge during drying and shaking or tapping the excess moisture from the bottom of the cartridge. Drying times are approximate. Do not over dry. Low recoveries could result

*For complete details on Method 8081B "Organochlorine pesticides by Gas Chromatography/Mass Spectrometry," December 1996,and 8082A, Polychlorinated Biphenyls by Gas Chromatography," the analyst is referred to Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

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