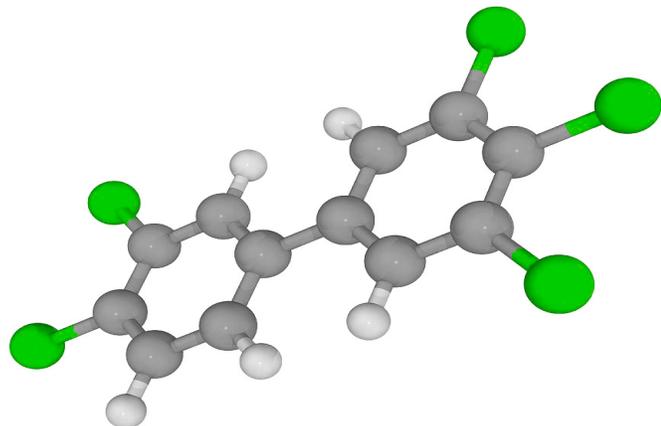


# Alternative Test Procedure for the Measurement of Organochlorine Pesticides and Polychlorinated Biphenyls in Waste Water - EPA Method 608 ATP\*



## UCT Part Numbers

### ECUNIC18

1100 mg endcapped C18, 83 mL

### Optional

### ECSS15M6

5000 mg Na<sub>2</sub>SO<sub>4</sub>, 6 mL

### EUFLSA1M6

1000 mg Grade A Florisil®, 6 mL

### ECCU01K

1 kg activated copper granules

### ECSS15M6

anhydrous. sodium sulfate  
drying tube

### EUFLS1M6

1000 mg PR Grade Florisil®, 6 mL

## Summary:

This is a gas chromatographic (GC) method for determination of compounds listed below in municipal and industrial discharges. The EPA has approved the use of C18 cartridges for this method.

## Analytes Recovered Using Method 608 ATP

Analyte	CAS
Aldrin	309-00-2
α-BHC	319-84-6
β-BHC	319-85-7
γ-BHC (Lindane)	58-89-9
δ-BHC	319-86-8
α-chlordane	5103-71-9
γ-chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
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
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2
PCB-1016	12674-11-2
PCB-1221	1104-28-2
PCB-1232	11141-16-5
PCB-1242	53469-21-9
PCB-1248	12672-29-6
PCB-1254	11097-69-1
PCB-1260	11096-82-5



## Procedure:

### 1. Condition Cartridge

- a) Insert cartridge(s) into the manifold or automated extraction system
- b) Wash with 10 mL of methylene chloride (MeCl<sub>2</sub>)
- c) Soak for 1 minute then draw through to waste
- d) Draw air under full vacuum to dry cartridge
- e) Add 10 mL of methanol (MeOH) then slowly draw through to top of frit
- f) Soak for 1 minute

**Note:** Do not let the cartridge go dry after addition of methanol otherwise repeat at step 1) e)

- g) Rinse the cartridge with 10 mL of reagent water
- h) Draw through leaving a thin layer on top of frit

### 2. Sample Extraction

- a) Adjust 1 liter of sample pH to < 2 using sulfuric acid
- b) If sample water is high in suspended solids, allow particulates to settle then slowly decant the water in the bottle. Once most of the water passes through the cartridge add the solids portion
- c) Draw the sample water through the cartridge over a 20-30 minute time period (fast drip) by adjusting the vacuum
- d) Dry the cartridge by drawing air under full vacuum through for 10 minutes

### 3. Extraction Elution

- a) Insert a collection tube into the vacuum manifold
- b) Add 5 mL of acetone to the sample bottle then swirl
- c) Add this to the cartridge
- d) Soak for 1 minute and slowly collect eluate
- e) Add 20 of methylene chloride to the sample bottle cover and shake. Add this to the cartridge
- f) Soak for 2 minutes and slowly collect eluate
- g) Rinse the inside walls of the sample bottle using 10 mL of methylene chloride then transfer solvent to the cartridge using a disposable pipette rinsing the inside of the cartridge
- h) Soak for 2 minutes then collect eluate

### 4. Sample Drying

- a) Pour the combined elutes together through a drying tube **ECSS15M6**. Alternatively, use 5 grams of sodium sulfate over a bed of glass wool in a funnel
- b) Rinse the drying tube or sodium sulfate bed with 2 x 3 mL portions of 1 methylene chloride
- c) Concentrate sample using a Kuderna-Danish (KD) concentrator while performing solvent exchange into hexane
- d) Concentrate sample under a gentle stream of N<sub>2</sub> while gently heating in a water bath. **Other drying techniques may be used**
- e) Rinse the inside walls of the concentrator tube two or three times with hexane during the evaporation
- f) Adjust the final volume of the extract to 10 mLs



## Florisil PR® or Copper Granule Clean-up Procedure (if needed)

Clean-up procedures may not be needed for relatively clean samples. If required, the following procedure can be used to remove polar interferences from organochlorine pesticide and PCB extracts in hexane eluants prior to analysis.

### 5. Florisil PR® Clean-Up

- a) Place a cartridge in a vacuum manifold
- b) Pre-rinse the Florisil® column with 10 mL of 90:10 hexane/acetone using gravity flow  
(a low vacuum may be necessary to start flow)
- c) Discard solvent
- d) Add a collection tube under the column
- e) Add a 2 mL aliquot of the sample extract (in hexane) to the cartridge
- f) Collect extract by gravity
- g) Add 10 mL of 90:10 hexane/acetone to the cartridge
- h) Continue to collect by gravity or low vacuum
- i) Gently evaporate the extract to a volume of 1 mL
- j) Adjust eluate to a final volume of 2 mL with hexane
- k) Sample is now ready for analysis

### 6. Sulfur Clean-up

- a) Place 4 grams of ECCU01K copper granules in a glass vial
- b) Add 2 mL of liquid sample extract to the vial
- c) Seal the glass vial and mix sample with copper for 2 minutes
- d) Allow to stand for approximately 10 minutes
- e) If sample contains high levels of sulfur, repeat process with 4 grams of fresh copper granules

**Note:** For the analysis of PCB type analytes, copper may reside in the extract

### 7. Analysis--GC/ECD

- a) Transfer clean extract to autosampler vial
- b) Sample is now ready for analysis



## References:

[1] \*The EPA has accepted the use of C18 bonded phases in packed cartridge format expanding the method from a disk only approach. For complete details on Method 607ATP, the analyst is referred to: "An alternative test procedure for the measurement of organochlorine pesticides and polychlorinated biphenyls in waste water", Federal register/Vol.60, No.148, August 2, 1995, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

Florisil® is a registered trademark of U.S. Silica

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UCT, LLC • 2731 Bartram Road • Bristol, PA 19007 800.385.3153 • 215.781.9255

[www.unitedchem.com](http://www.unitedchem.com) Email: [methods@unitedchem.com](mailto:methods@unitedchem.com)

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