



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

UCT Product Numbers:

**ECUNIC18** - 1100 mg endcapped C18, 83 mL

**ECUNIMSS** - Enviro Clean Universal Cartridge 20g Muffled Sodium Sulfate

**ECCU01K** - 1 kg activated copper granules

**EUFLS1M6** - 1000 mg PR Grade Florisil®, 6 mL

**EUFLSA1M6** - 1000 mg Grade A Florisil®, 6 mL

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 SPE for this method.

### Analytes Recovered Using Method 608ATP

Analyte	CAS
Aldrin	309-00-2
$\alpha$ -BHC	319-84-6
$\beta$ -BHC	319-85-7
$\gamma$ -BHC (Lindane)	58-89-9
$\delta$ -BHC	319-86-8
$\alpha$ -chlordane	5103-71-9
$\gamma$ -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 ( $\text{MeCl}_2$ )
- c) Soak for 1 min then draw through to waste
- d) Draw air under full vacuum to dry cartridge
- e) Add 10 mL of methanol ( $\text{MeOH}$ ) then slowly draw through to top of frit
- f) Soak for 1 min

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 min

### 3. Extract 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 min and slowly collect eluate
- e) Add 20 mL of methylene chloride to the sample bottle cover and shake. Add this to the cartridge
- f) Soak for 2 min and slowly collect the 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 min then collect eluate

#### 4. Sample Drying

- a) Pour the combined elutes together through a drying cartridge (ECUNIMSS) pre-rinsed with methylene chloride. Alternatively, use 15-20 grams of sodium sulfate over a bed of glass wool in a glass funnel
- b) Rinse the eluate collection tube with 2 x 5 mL of methylene chloride, apply the rinse to the sodium sulfate bed and collect
- 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

#### Florisol PR<sup>®</sup> 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. Florisol PR<sup>®</sup> Clean-Up

- a) Place a cartridge in a vacuum manifold
- b) Pre-rinse the Florisol<sup>®</sup> 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 min
- d) Allow to stand for approximately 10 min
- 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;  
Copper cleanup may result in low recoveries for several pesticides.**

## 7. Analysis--GC/ECD

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

\*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

5108-10-01