



**ENVIRO-CLEAN® Cartridge**  
**EPA Method 608 ATP\***  
Part #: ECUNIC18

The EPA has accepted the use of C18 bonded phases in packed cartridge format expanding the method from a disk only approach. This method is used in place of liquid-liquid extraction. The UCT ECUNIC18 Universal cartridge has been designed to provide a high level of performance for the solid-phase extraction (SPE) and analysis of certain **organochlorine pesticides and PCB's** in municipal and industrial discharges. With the cartridge's high capture efficiency, fast flow and rapid dry times, laboratory throughput can be significantly improved and sample preparation time reduced.

### **Product Benefits**

- SPE cartridge containing bonded C18 phase
- Excellent pH stability under acidic conditions
- Fast flow rates for rapid analyte capture
- Works well at all levels of analyte loading
- Consistent results for excellent reproducibility
- PTFE frits eliminate potential contamination yielding clean extracts
- Packaged in metalized bags to maintain product cleanliness

### **Product Features**

- Cartridges manufactured from clean proprietary polypropylene
- Each cartridge contains 1100 mg endcapped C18 bonded ultra-clean silica sorbent
- Can be used on manual single or multi-station vacuum manifold systems
- Can be used with a variety of automated extraction systems

## UCT Products Required:

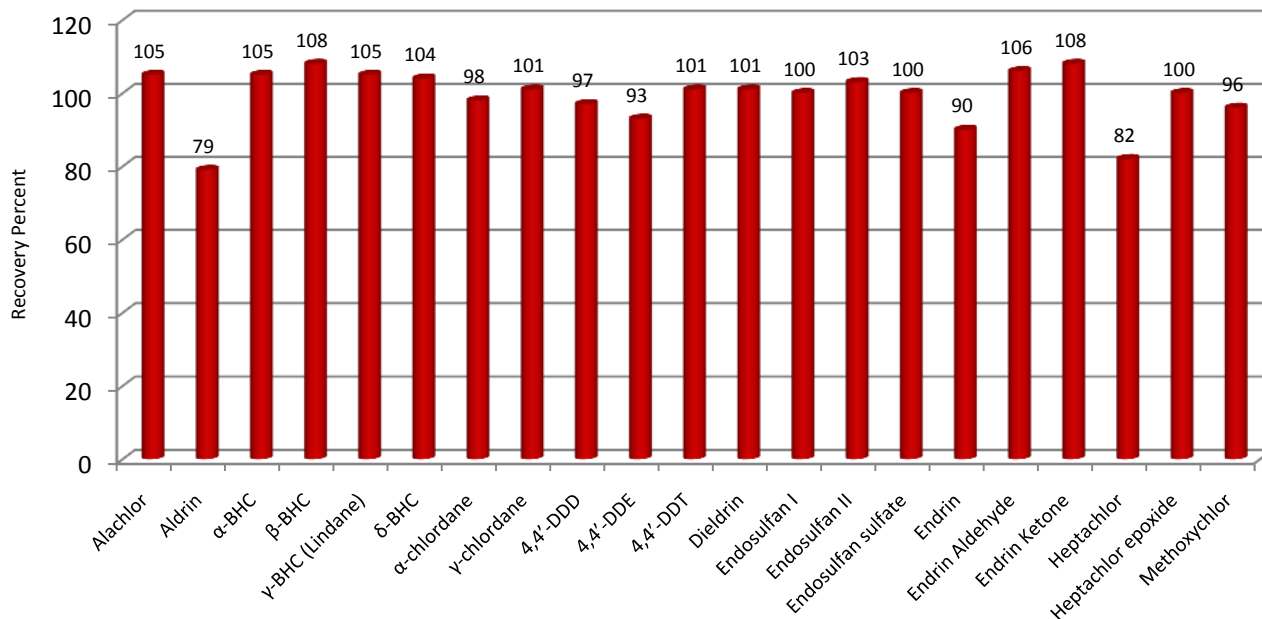
- 1) Universal Cartridge ECUNIC18
- 2) Florisil PR<sup>®</sup> column cleanup (optional) EUFLS1M6
- 3) NaSO<sub>4</sub> Drying Tube UCT ECSS15M6

## Recovery of Method 608ATP Analytes

Analyte	CAS	Amt Spiked µg/L	Average Recovery %	Stdev
Aldrin	309-00-2	0.0999	79	2.08
α-BHC	319-84-6	0.1032	105	0.96
β-BHC	319-85-7	0.1043	108	1.50
γ-BHC (Lindane)	58-89-9	0.1038	105	0.82
δ-BHC	319-86-8	0.1040	104	1.26
α-chlordane	5103-71-9	0.0969	98	0.50
γ-chlordane	5103-74-2	0.0969	101	1.71
4,4'-DDD	72-54-8	0.2056	97	2.08
4,4'-DDE	72-55-9	0.2006	93	2.38
4,4'-DDT	50-29-3	0.2014	101	0.96
Dieldrin	60-57-1	0.2046	101	0.82
Endosulfan I	959-98-8	0.1028	100	0.82
Endosulfan II	33213-65-9	0.2054	103	0.96
Endosulfan sulfate	1031-07-8	0.2124	100	2.06
Endrin	72-20-8	0.2016	90	7.33
Endrin Aldehyde	7421-93-4	0.2012	106	9.54
Endrin Ketone	53494-70-5	0.2068	108	1.73
Heptachlor	76-44-8	0.1032	82	5.29
Heptachlor epoxide	1024-57-3	0.1034	100	0.96
Methoxychlor	72-43-5	1.0016	96	1.71

**UCT Cartridge ECUNIC18 Shows Excellent Recovery with  
Laboratory Fortified Blanks (LFB)**

## Recovery of Chlorinated Pesticides & Herbicides by Method 608ATP



## An Alternative Test Procedure for the Measurement of Organochlorine Pesticides and Polychlorinated Biphenyls in Waste Water

### Method 608ATP

Federal Register Vol. 60, # 148, August 2, 1995

### Method Summary\*

A 1-liter sample of water is extracted by drawing through a **UCT C18 Universal cartridge ECUNIC18**. The analytes captured on the solid-phase are eluted from the cartridge using a small volume of acetone followed by methylene chloride ( $\text{MeCl}_2$ ). The eluant is dried and exchanged into hexane for analysis by injection into a gas chromatograph with electron capture detection system (GC/ECD) fitted with a high-resolution fused silica capillary column.

## Interferences

- Interferences may generally be attributed to contamination from solvents, glassware or other laboratory equipment leading to anomalous GC peaks. Glassware must be scrupulously cleaned and high purity solvents used.
- Interfering contamination may occur when a sample of low concentration is analyzed immediately after a sample of high concentration. A laboratory blank should be inserted between low and high concentration samples to minimize this potential problem
- Phthalate esters may pose a problem in pesticide analysis when using electron capture detection. This results from contact with common laboratory plastics such as PVC

## Sample Collection

- Samples must be collected in glass containers following conventional practices **except** the bottle must not be prerinsed with sample before collection
- Samples must be refrigerated at 4 °C from collection to analysis and extracted within 72 hours of collection
- If samples are to be held longer than 72 hours the pH must be adjusted to 5.0-9.0 with sodium hydroxide or sulfuric acid depending upon initial pH

## Procedure

### 1) Condition Cartridge

- a) Insert a cartridge into the glass vacuum manifold or automated extraction system
- b) Wash the cartridge with 10 mL of methylene chloride (MeCl<sub>2</sub>). Apply solvent for 1 minute then draw through to waste
- c) Draw air under full vacuum to completely dry cartridge
- d) Add 10 mL of methanol (MeOH) to the cartridge then slowly draw some of it through
- e) Allow the cartridge to soak for 1 minute in methanol
- f) Do not let the cartridge go dry after addition of methanol otherwise repeat the addition of methanol addition step
- g) Rinse the cartridge with 10 mL of reagent water and draw most of it through leaving a thin layer on the top of the sorbent

## 2) Sample Addition

- a) Adjust sample pH to < 2 using sulfuric acid
- b) Adjust the vacuum and draw the sample water through the cartridge over a 20-30 minute time period
- c) 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
- d) Dry the cartridge by drawing air through for about 5-10 minutes

## 3) Extract Elution

- a) Insert an eluate 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 (**UCT ECSS15M6**) which contains 5 grams anhydrous sodium sulfate. 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. Other drying techniques may be used
- d) Concentrate sample under a gentle stream of N<sub>2</sub> while gently heating in a water bath
- 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<sup>®</sup> Clean-up (if needed)**

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

**UCT EUFLSA1M6** – 1000 mg small particle Grade A Florisil<sup>®</sup> for slower gravity flow

**UCT EUFLS1M6** – 1000 mg regular particle PR Grade Florisil<sup>®</sup> for more viscous samples

### **1) Procedure**

- a) Place a cartridge in a vacuum manifold
- b) Prerinse the Florisil<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 column
- f) Collect extract by gravity
- g) Add 10 mL of 90:10 hexane/acetone to the column
- 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

## Sulfur Clean-up (if needed)

UCT ECCU01K – 1 kg copper granules

### 1) Procedure

#### a) Post Sample Extraction

- a) Place 4 grams of copper bead in a glass vial
- b) Add 2 mL of liquid sample extract to the vial

#### b) Sulfur Removal

- a) Seal the glass vial and mix sample with copper beads for 2 minutes
- b) Allow to stand for approximately 10 minutes
- c) If sample contains high levels of sulfur, repeat process with 4grams of fresh copper beads

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

#### c) Analysis, GC/MS or LC/MS

- a) Transfer clean extract to autosample vial
- b) Inject 1-2  $\mu$ L for GC
- c) Inject 5-10  $\mu$ L for LC

#### d) Sample is now ready for 6081tp analysis

## Sample Analysis by 608ATP

### 5) Analysis

- a) Inject a 1-2  $\mu$ L aliquot into a GC
- b) Identify the analytes in the sample by comparison of the retention time to known reference chromatograms

\*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<sup>®</sup> is a registered trademark of U.S. Silica

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