ENVIRO

UCT

# Clean-Up and Fractionation

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# Clean-Up of Organochlorine Pesticides and PCB Extracts Using FLORISIL®

Part #: EUFLSA1M6 or EUFLS1M6

February 3, 2009

This application is designed to remove polar interferences from organochlorine pesticide and PCB extracts in hexane prior to analysis.

## REAGENTS:

Hexane
Acetone

## Product Description:

EUFLSA1M6 – 1000 mg small particle Grade A Florisil® for slower gravity flow.
EUFLS1M6 – 1000 mg regular particle PR Grade Florisil® for more viscous samples.

## PROCEDURE:

1. Prerinse a column with 9 mL of 90:10 hexane/acetone by gravity. (A low vacuum may be necessary to start flow)
2. Discard solvent.
3. Add a collection tube under the column.
4. Add a 2 mL aliquot of the sample extract (in hexane) to the column.
5. Collect extract by gravity.
6. Add 9 mL of 90:10 hexane/acetone to the column.
7. Continue to collect by gravity.
8. Gently evaporate the extract to a volume of 1 mL.
9. Bring to a final volume of 2 mL with hexane.

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



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# Fractionation of Aliphatic And Aromatic Hydrocarbons Using ENVIRO-CLEAN® EPH SILICA

(Developed in cooperation with Lancaster Laboratories, Inc.)

**UCT Part Number:** XRSIHT13M15

February 3, 2009

**Background:**

The composition of petroleum is a complex mixture of hundreds of different hydrocarbon compounds. The resultant makeup of hydrocarbons released into the environment is variable and dependent on the conditions to which it is subsequently exposed. While in the environment, petroleum composition is influenced by a number of factors including volatilization, leaching and/or biological degradation. These environmental effects yield a mixture whose toxicological properties can be vastly different than the parent product. Based on the known toxicological properties of petroleum products we can assume that:

- aromatic compounds are more toxic than aliphatic compounds
- the toxicity of aliphatic compounds is dependent upon their molecular weight with low molecular weight compounds showing relatively higher toxicity

The fractionation of the total petroleum hydrocarbon extract is necessary to determine the concentration of the aliphatic versus aromatic compounds. The Massachusetts Department of Environmental Protection (MADEP) has taken the approach of fractionating the C9-C18 aliphatics (n-nonane to n-octadecane), C19-C36 aliphatics (n-nonadecane to hexatriacontane), and the C11-C22 aromatics (naphthalene to benzo (g,h,i)perylene). These compounds are associated with the release of hydrocarbons in the environment. The aromatics are considered the most toxic form of hydrocarbon.

**Procedure:**

**Prepare Extract**

1. Solvent exchange the hydrocarbon extract from methylene chloride to hexane using a K-D apparatus.

**Prepare Cartridge**

2. Thoroughly rinse cartridge with two, 10 mL aliquots of pentane.

3. Add 1 mL of the extract to the cartridge.

4. Elute aliphatic fraction with pentane by gravity and collect everything in an ampoule. A total of 10 mL should be collected.

5. Place a fresh ampoule under the cartridge and elute the aromatic fraction with methylene chloride by gravity. A total of 10 mL should be collected.

6. Concentrate each fraction separately to a final volume on a steam bath using an ampoule and micro-Snyder column combination. Other techniques may be used but the loss of C9-C18 hydrocarbons may result.

It is very important to keep the silica cartridges dry and away from room air prior to use. Moisture and contaminants in the air will reduce the effectiveness of the silica and may cause contamination of the extract. Pre-rinsing the cartridges with acetone may reduce this problem.

# Results:

| Classification | Range | Percent Recovery |
|---|---|---|
| **Aromatics** | **C11-C22** | **88** |
| surrogates | 2-fluorobiphenyl | **123** |
| surrogates | o-terphenyl | **100** |
| **Aliphatics** | **C9-C18** | **85** |
| | **C19-C36** | **89** |
| surrogates | 1-chlorooctadecane | **58** |

MA EPH DATA from Lancaster Labs

**UCT in cooperation with Lancaster Laboratories, Inc., has developed a fractionation product that provides consistent and accurate results free from contamination**



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# Removal of Sulfur from Environmental Samples Using Copper Beads

**UCT Part Number:** ECCU01K

February 24, 2009

1. **Post Sample Extraction**
   a. Place 4 grams of copper beads in a glass vial
   b. Add 2 mL of liquid sample extract to the vial

2. **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 4 grams of fresh copper beads

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

3. **Analysis, GC/MS or LC/MS**
   a. Transfer clean extract to autosampler vial
   b. Inject 1-2 µL for GC
   c. Inject 5-10 µL for LC



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