Determination of Phthalate and Adipate Esters in Drinking Water by Liquid-Solid Extraction and Gas Chromatography with Photoionization Detection

UCT Part Numbers:
ECUNIC18 (1100 mg C18, 83 mL cartridge) or **EEC181M6G** (1000 mg C18, 6 mL glass cartridge)
ECSS156 (Drying Cartridge 5 g, Na_2SO_4, 6 mL cartridge)
FLORISIL PR® Clean-up Cartridge EUFLS12M15 (2000 mg,15 mL cartridge)
Alumina Clean-up Cartridge EUALN1M6 (1000 mg, 6 mL cartridge)
ENVIRO-CLEAN® Zero-Blank™ filter cartridge (optional)

EPA Method 506 Revison 1.1*

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Procedure

1. Cartridge Activation

a) Place **ECUNIC18** cartridge(s) on the manifold
b) Add a 10 mL aliquot of methylene chloride
c) Slowly draw solvent completely through the cartridge
d) Repeat with a second 10 mL aliquot of methylene chloride
e) Hold for 1 minute then draw through
f) Dry cartridge by drawing air at full vacuum for 2-3 minutes

Optional: To reduce background contamination from laboratory air, use of the **ENVIRO-CLEAN® Zero-Blank™** filter cartridge is highly recommended during drying steps 1) f) and 2) c)

g) Add 10 mL of methanol to the cartridge
h) Draw through leaving a thin layer on the frit

Note: Do not allow the cartridge to go dry otherwise repeat step i

i) Add a second 10 mL portion of methanol
j) Wait 1-2 minutes to activate sorbent then draw through to level of frit
k) Add 10 mL reagent water to the cartridge and draw through until meniscus reaches the top of the frit
l) Cartridge is now ready for sample extraction

2. Sample Extraction

a) Add the 1 liter water sample to the cartridge and draw through over a period of about 20–30 minutes (fast drip)

b) Rinse sample bottle and cartridge with a small volume of reagent water then add to cartridge

c) Dry cartridge under full vacuum for 10 minutes (**Optional**: Use **Zero-Blank™** filter cartridge to prevent airborne contamination)

3. Cartridge Elution

a) Place a collection vial in the vacuum manifold

b) Add 5 mL of methylene chloride to the sample bottle and swirl

c) Using a disposable glass pipette transfer the methylene chloride to the cartridge

d) Collect dropwise

e) Repeat this procedure with another 5 mL of methylene chloride

4. Eluate Drying and Concentration

a) Pour eluant through a 3 gram bed of anhydrous sodium sulfate (or use Drying Cartridge **ECSS156**) and collect

b) Rinse vial and sodium sulfate with a 3 mL aliquot of methylene chloride

c) Repeat rinse using an additional 3 mL aliquot of methylene chloride

d) Evaporate with a gentle stream of N_2 to 1 mL

e) If sample is clean proceed to GC analysis

f) If extract requires clean-up for phthalates esters proceed to Florisil or Alumina clean-up stages

Extract Clean-up---Florisil or Alumina

Clean-up procedures are not required for clean drinking water. Under certain circumstances for dirty water, a Florisil or Alumina clean-up may be needed.

Florisil Column Clean-up for Phthalate Esters

1. Add a 1 cm layer of anhydrous sodium sulfate to the top frit of Florisil cartridge **EUFLS12M15**
2. Flush cartridge with 20 mL of hexane leaving enough to cover the frit
3. Add sample extract to the cartridge then rinse vial with 2 mL of hexane
4. Add 20 mL of hexane to the cartridge and elute. Discard the hexane solution

5. Elute using 20 mL of 20% diethyl ether in hexane into a 500 mL K-D flask* equipped with a 10 mL concentrator tube. Elute at a rate of about 2 mL/minute
6. No solvent exchange is required
7. Concentrate eluate in hot water bath at 85° C to 1 mL
8. Sample is ready for GC analysis

Alumina Column Clean-up (neutral) for Phthalate Esters

1. Add a 1 cm layer of anhydrous sodium sulfate to the top frit of a Alumina cartridge **EUALN1M6**
2. Flush cartridge with 10 mL of hexane leaving enough to cover sodium sulfate
3. Add sample extract to the cartridge. Rinse vial with 2 mL of hexane and add to the cartridge
4. Add 15 mL of hexane to the cartridge and elute. Discard hexane solution
5. Elute at a rate of about 2 mL/minute using 15 mL of 20% diethyl ether in hexane
6. Collect eluate in a 500 mL K-D flask equipped with a 10 mL concentrator tube
7. No solvent exchange is required
8. Concentrate eluate in hot water bath at 85° C to 1 mL
9. Sample is ready for GC analysis

*The analyst should refer to EPA Method 506 “Determination of Phthalate and Adipate Esters in Drinking Water by Liquid-Solid Extraction and Gas Chromatography with Photoionization Detection”, Revision 1.1 Issued 1995, F.K. Kawahara and J.W. Hodgeson, Ed. By D. J. Munch US EPA, National Exposure Research Laboratory, Office of Research and Development, US Environmental Protection Agency, Cincinnati, Ohio 45268
**K-D Flask = Kuderna-Danish Flask