EPA Method 8270: Determination of Semi-volatile Organic Compounds in Water using Solid Phase Extraction and GC/MS

UCT Part Numbers:
EC82701M15 - 1000 mg 8270 sorbent/15 mL cartridge (≤ 500 mL sample)
EU52112M6 - 2000 mg activated carbon/6 mL cartridge (≤ 500 mL sample)
EC82702M15 - 2000 mg 8270 sorbent/15 mL cartridge (> 500 mL sample)
EU52113M6 - 3000 mg activated carbon/6 mL cartridge (> 500 mL sample)
AD0000AS - Cartridge adaptor
VMFSTFR12 - Large volume sample transfer tubes
VMF016GL - 16 position glass block manifold
VMF02125 - 12 position large volume collection rack
RFV1F15P - 15 mL reservoirs with 1 frit, 10 micron porosity
ECSS25K - Sodium sulfate, anhydrous, ACS grade, granular, 60 mesh
GCLGN4MM-5 - GC liner, 4 mm splitless gooseneck

EPA method 8270 allows the use of liquid-liquid extraction (LLE) and solid phase extraction (SPE) to extract semi-volatile organic compounds (SVOCs) in aqueous samples and TCLP leachates. LLE requires multiple extractions at two different pH values, consumes large amounts of organic solvents, and causes emulsion when real world dirty samples are extracted.

This application note outlines a reliable, efficient, and cost-effective SPE method utilizing two stacked SPE cartridges, UCT's EC8270 and activated carbon cartridges for the extraction of SVOCs in water and TCLP samples. Prior to extraction, samples are dechlorinated and adjusted to pH < 2, then passed through the SPE system, the 8270 SPE cartridge retains the majority of the target analytes including acids, bases, and neutrals with mid to high hydrophobicity, while the carbon cartridge connected downstream will capture a few very polar compounds*, such as *1,4-dioxane*, n-nitrosodimethylamine, n-nitrosomethylethylamine, methyl methanesulfonate, ethyl methanesulfonate, and1-Nitrosopyrrolidine. High sample throughout can be achieved by extracting multiple samples simultaneously using a multi-port SPE manifold.

**: Carbon cartridge is NOT needed if none of the very polar analytes is being analyzed, such as for the TCLP SVOCs list.*

SPE Procedure:

1. Sample Pretreatment

a) Dechlorinate sample with 80 mg/L of sodium thiosulfate if free chlorine presents.
b) Adjust sample pH to < 2 using 6N HCl.
c) Spike with surrogates, and target analytes for fortified samples.

Tip 1: The spiking solutions should be prepared in water miscible solvents, such as methanol, acetonitrile, or acetone.

2. SPE System Setup

a) Connect the carbon cartridge (**EU52112M6** or **EU52113M6** depending on sample volume) to the end of the 8270 cartridge (**EC82701M15** or **EC82702M15** depending on sample volume) using a cartridge adaptor (**AD0000AS**).
b) Insert a loose plug of deactivated glass wool into the 8270 cartridge to prevent sorbent clogging caused by samples with high particulate content.
c) Attach the connected SPE cartridges to the SPE manifold (**VMF016GL**).

Tip 2: The carbon cartridge is not needed if very polar analytes, such as 1,4-dioxane, n-nitrosodimethylamine, n-nitrosomethylethylamine, methyl methanesulfonate, ethyl methanesulfonate, and 1-Nitrosopyrrolidine are not being analyzed.

3. Cartridge Conditioning

a) Wash the SPE cartridges with 15 mL of dichloromethane (DCM), soak 1 min, and apply full vacuum for 1 min.
b) Condition the SPE cartridges with 10 mL of methanol. Draw most of the way through the column leaving a thin layer (about 0.5 cm) of solvent above the frit. Do not allow cartridges to go dry from this step until instructed to do so in the cartridge drying step.
c) Equilibrate the cartridges with 10 mL of reagent water and 10 mL of 0.05N HCl.

4. Sample Loading

a) Attach the large volume sample delivery tube (**VMFSTFR12**) to the top of the 8270 cartridge, and insert the stainless steel end of the tube into the sample bottle.

b) Adjust vacuum for a fast dropwise sample flow (about 10-15 mL/min), and draw the entire sample through.

5. Washing and Drying

a) Rinse the sample bottle with 10 mL of reagent water, and apply the rinsate to the SPE cartridges.

b) Disassemble the transfer tube and the connected SPE cartridges. Dry the 8270 cartridge under full vacuum for 10 min, and the carbon cartridge for 15 min.

Tip 3: Remove as much water as possible, wet sorbents result in low analyte recovery.

6. Analyte Elution

a) Insert the collection rack (**VMF02125**) with 40-60 mL glass vials into the manifold.

b) Elute the SPE 8270 and carbon cartridges separately. Apply elution solvent to the SPE cartridges, draw 1/3 through, soak 1-2 min, and then draw the remaining solvent through the cartridge in a slow dropwise fashion. Leave full vacuum on for 1 min after each elution.

8270 Cartridge	10 mL 1:1 acetone:n-hexane (bottle rinse added to 8270 cartridge using transfer tube)
	10 mL DCM
	3 - 4 mL ammonium hydroxide (28-30%), drain to waste
	3 x 7 mL DCM
Carbon Cartridge	5 x 3 mL DCM

Tip 4: Bottle rinse is critical for good recovery of PAHs, which tend to adsorb on the glass wall.

7. Eluate Drying

a) Dry the eluates using a 15-mL reservoir (or a glass funnel stopped with glass wool) holding about 15-20 g of anhydrous Na_2SO_4, pre-rinse the Na_2SO_4 with 10 mL of DCM.

b) Insert the collection rack with 40-60 mL glass vials into the manifold to collect the dried eluates.

c) Pass the eluates through the Na_2SO_4 bed.

d) Rinse the eluate vials with 2 x 5 mL of DCM, transfer the rinses to the Na_2SO_4 bed.

Tip 5: If Na_2SO_4 appears greenish, rinse with more solvent until it turns white.

8. Concentration

a) Concentrate the eluates to 0.7-0.9 mL under a gentle stream of N_2 at 40 ºC.

b) Add internal standards, transfer the extract to a 2-mL auto-sampler vial, and adjust the final volume to 1 mL.

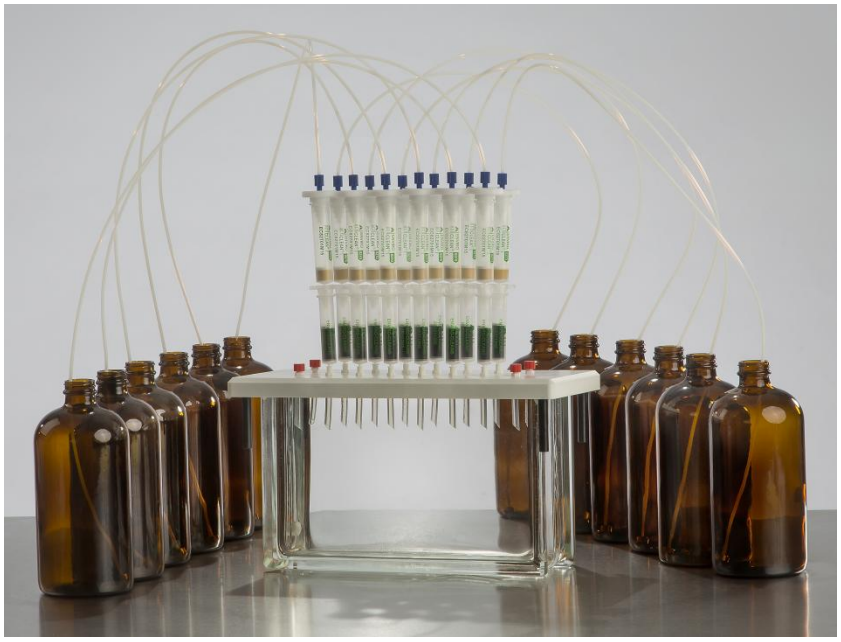
c) The samples are ready for GC/MS analysis.

GC/MS Method

GC/MS	Agilent 6890N GC coupled to a 5975C MSD
Injection	1 µL splitless injection at 250 ºC, split vent of 30 mL/min at 1 min
GC Liner	4 mm splitless gooseneck (GCLGN4MM-5), packed with deactivated glass wool
GC Column	Restek Rxi®-5sil MS 30m x 0.25mm, 0.25µm with 10m integrated guard column
Carrier Gas	Ultra high purity helium at a constant flow of 1.5 mL/min
Oven Temp. Program	Initial temperature at 40 ºC, hold for 3 min; ramp at 15 ºC/min to 240 ºC; ramp at 6 ºC/min to 310 ºC; and hold for 2 min
MSD Temp.	Transfer line 280 ºC; Source 250 ºC; Quadrupole 150 ºC

Full Scan Range	35 - 500 amu

SPE Setup



Recovery and RSD in Laboratory Fortified Blanks

(500 mL sample fortified with 40 µg/L of 139 analytes and 6 surrogates)

Compound	Ave Recovery%	RSD% (n=4)
1,2,4,5-Tetrachlorobenzene	**99.5**	4.1
1,2,4-Trichlorobenzene	**88.5**	5.7
1,2-Dichlorobenzene	**90.3**	3.9
1,3,5-Trinitrobenzne	**124.4**	2.8
1,3-Dichlorobenzene	**85.8**	2.8
1,4-Dichlorobenzene	**89.1**	1.1
1,4-Naphthalenedione	**95.3**	4.3
1-Chloronaphthalene	**112.2**	2.7
1-Methyl fluorene	**86.9**	0.9
1-Methyl phenanthrene	**89.8**	1.3
1-Methylnaphthalene	**102.1**	2.7
1-Naphthalenamine	**112.3**	4.7
1-Nitrosopiperidine	**88.9**	5.8
1-Nitrosopyrrolidine	**91.8**	7.2

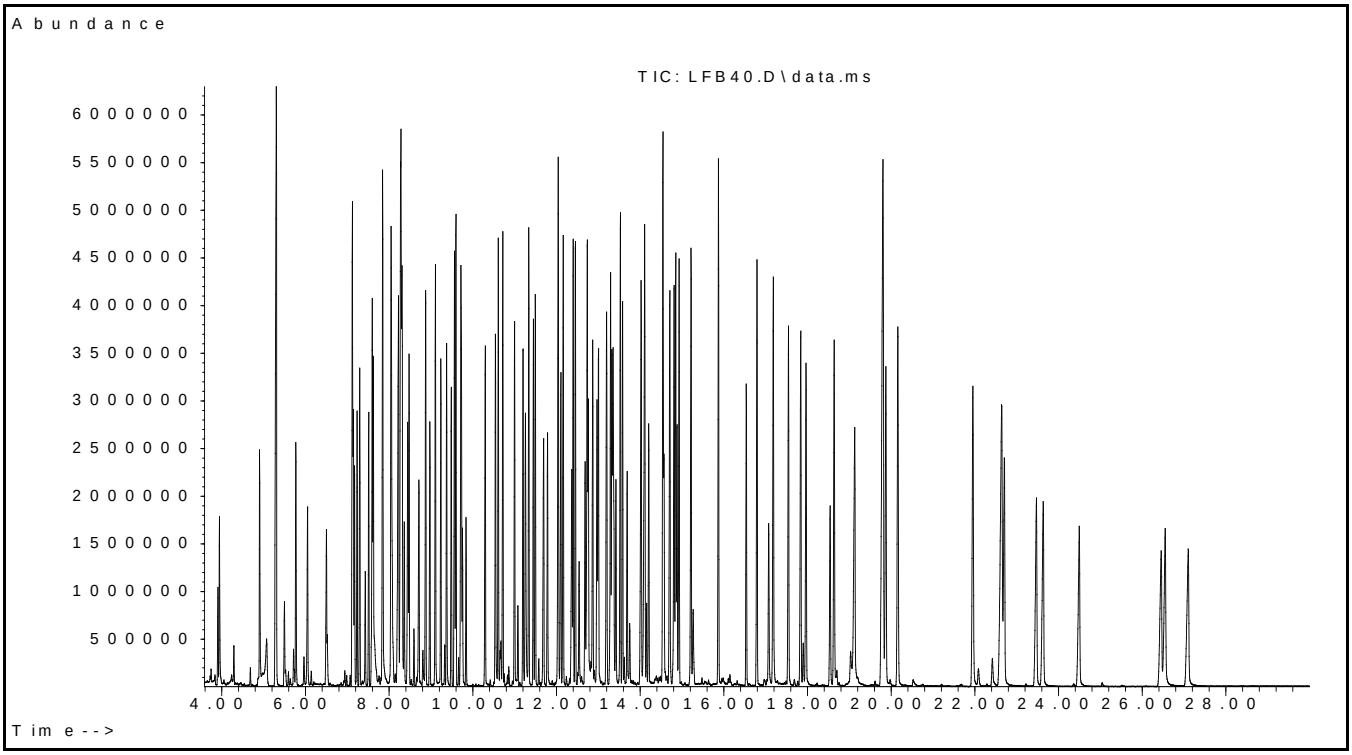
2,3,4,6-Tetrachlorophenol	**103.2**	0.9
2,3-Dichloroaniline	**91.4**	0.6
2,4,5-Trichlorophenol	**123.5**	4.7
2,4,6-Trichlorophenol	**106.5**	3.6
2,4-Dichlorophenol	**97.3**	6.5
2,4-Dimethylphenol	**99.0**	6.4
2,4-Dinitrophenol	**122.4**	2.0
2,4-Dinitrotulene	**112.0**	1.7
2,6-Dichlorophenol	**113.3**	0.7
2,6-Dinitrotoluene	**106.3**	2.3
2-Acetylaminofluorene	**109.0**	6.5
2-Chloronaphthalene	**96.9**	2.8
2-Chlorophenol	**99.4**	2.9
2-Isopropyl naphthalene	**73.1**	0.1
2-Methylnaphthalene	**101.2**	4.9
2-Methylphenol	**97.6**	6.7
2-Naphthalenamine	**130.5**	2.7
2-Nitroaniline	**107.5**	3.6
2-Nitrophenol	**98.2**	5.9
2-Picoline	**74.4**	5.0
3&4-Methylphenol	**104.2**	6.6
3,3'-Dichlorobenzidine	**72.3**	11.4
3,6-Dimethyl phenanthrene	**90.6**	0.9
3-Methylcholanthrene	**106.5**	1.4
3-Nitroaniline	**100.4**	4.9
3-Nitrophenol	**99.5**	8.2
4,4'-DDD	**94.4**	0.8
4,4'-DDE	**91.8**	0.4
4,4'-DDT	**94.0**	0.3
4,6-Dinitro-2-methylphenol	**116.8**	4.5
4-Aminobiphenyl	**103.8**	13.5
4-Chloro-3-methylphenol	**111.7**	6.3
4-Chloroaniline	**105.0**	3.9
4-Chlorophenylphenylether	**99.5**	3.0
4-Nitroaniline	**114.9**	4.6
4-Nitrophenol	**97.2**	3.0
5-Nitro-o-toluidine	**94.7**	4.0
7,12-Dimethyl benz[a]anthracene	**99.9**	6.1
Acenaphthene	**100.1**	1.3
Acenaphthylene	**102.6**	0.6
Acetophenone	**101.8**	7.4

Aldrin	**89.5**	0.8
alpha lindane	**90.1**	0.2
Aniline	**90.0**	3.2
Anthracene	**109.7**	1.1
Azobenzene	**105.5**	5.2
Benz[a]anthracene	**103.3**	6.2
Benzidine	**66.8**	14.0
Benzo[a]pyrene	**99.3**	2.1
Benzo[b]fluoranthene	**99.4**	7.0
Benzo[ghi]perylene	**104.2**	1.1
Benzo[k]fluoranthene	**108.1**	5.4
Benzoic acid	**115.0**	4.7
Benzyl alcohol	**72.9**	12.9
Benzyl butyl phthalate	**111.8**	6.0
beta lindane	**95.2**	1.1
Bis(2-ethylhexyl) phthalate	**113.2**	2.0
Bis[2-chloroethoxy]methane	**91.0**	7.8
Bis[2-chloroethyl]ether	**88.5**	3.0
Bis[2-chloroisopropyl]ether	**87.3**	4.5
Bromophenoxybenzene	**99.6**	4.8
Carbazole	**109.6**	3.3
Chlorobenzilate	**116.3**	9.4
Chrysene	**103.3**	1.2
delta lindane	**95.2**	0.8
Diallate (cis & trans)	**104.7**	4.5
Dibenz[ah]anthracene	**108.8**	2.5
Dibenzofuran	**102.0**	0.6
Dibutyl phthalate	**114.6**	6.2
Dieldrin	**94.5**	0.7
Diethyl phthalate	**110.4**	1.2
Dimethoate	**96.6**	0.7
Dimethyl phthalate	**110.3**	1.3
Di-n-octyl phthalate	**116.6**	5.9
Dinoseb	**121.9**	1.7
Diphenylamine	**109.9**	4.8
Disulfoton	**87.0**	0.6
Endosulfan I	**93.8**	0.7
Endosulfan II	**96.5**	0.5
Endosulfan sulfate	**96.2**	0.7
Endrin	**97.4**	1.0
Endrin aldehyde	**93.4**	0.5

Ethyl methanesulfonate	**92.5**	3.7
Famphur	**109.3**	1.2
Fluoranthene	**105.8**	6.1
Fluorene	**103.7**	2.6
gamma lindane	**93.1**	1.3
Heptachlor	**88.1**	1.0
Heptachlor epoxide	**93.4**	0.9
Hexachlorobenzene	**101.3**	6.1
Hexachlorobutadiene	**85.0**	1.0
Hexachloroethane	**92.6**	6.0
Hexachloropropene	**72.1**	1.1
Hexachorocyclopentadiene	**85.9**	3.1
Indeno[123-cd]pyrene	**103.2**	2.5
Isodrin	**105.1**	7.2
Isophorone	**91.0**	6.8
Isosafrole (cis & trans)	**102.9**	6.1
Methyl methanesulfonate	**70.8**	3.5
Methyl parathion	**96.6**	0.4
Naphthalene	**97.2**	2.3
Nitrobenzene	**94.0**	7.2
N-nitro-di-n-propylamine	**99.3**	6.3
N-nitroso di-n-butylamine	**99.9**	4.7
N-nitrosodiethylamine	**89.4**	3.7
N-nitrosodimethylamine	**68.8**	3.0
N-nitrosomethylethylamine	**87.4**	2.5
o,o,o-Triethylphosphorothioate	**90.8**	0.4
o-Toluidine	**91.4**	9.7
Parathion	**95.8**	0.7
p-Dimethylaminoazobenzene	**91.5**	10.5
Pentachlorobenzene	**90.9**	1.0
Pentachloroethane	**86.0**	3.8
Pentachloronitrobenzene	**104.3**	4.2
Pentachlorophenol	**109.3**	3.3
Phenacetin	**116.4**	3.9
Phenanthrene	**108.0**	0.4
Phenol	**56.2**	4.2
Phorate	**86.7**	0.1
Pronamide	**111.2**	5.2
Pyrene	**109.1**	8.5
Pyridine	**46.1**	8.0
Safrole	**90.7**	4.3

Sulfotep	**92.5**	0.8
Thionazin	**95.1**	0.7
Surrogates		
2-Fluorophenol (S)	**87.2**	0.6
Phenol d6 (S)	**59.1**	0.4
Nitrobenzene d5 (S)	**94.3**	1.0
2-Fluorobiphenyl (S)	**81.5**	0.5
2,4,6-Tribromophenol (S)	**95.4**	0.2
p-Terphenyl d14 (S)	**97.5**	1.0

Chromatogram of an LFB Fortified at 40 µg/L



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