Scaled Down EPA Methods 8270 and 625
Solid Phase Extraction of 100 mL Samples or Less

UCT Part Numbers:
EC8270506 - 500 mg EC8270 sorbent/6 mL cartridge
EU5211M6 - 1000 mg activated carbon/6 mL cartridge
AD0000AS - Cartridge adaptor
VMFSTFR12 - Large volume sample transfer tubes
VMF016GL - 16 port glass block vacuum manifold
VMF06125 - Collection rack set (16 port) - for 10 & 16mm test tubes
RFV1F15P - 15 mL reservoirs with 1 frit, 10 micron porosity
ECSS25K - Sodium sulfate, anhydrous, ACS grade, granular, 60 mesh

The EPA has updated methods 8270 (8270E) and 625 (625.1) to both now permit the use of solid phase extraction (SPE) to analyze for semi-volatile organic compounds (SVOCs) in aqueous samples and TCLP leachates. The revised methods will also now allow more sensitive and selective instrumentation for analyte detection and quantitation, such as GC/MS with large volume injection (e.g. PTV), GC/MS in SIM mode, ion trap MS, and tandem MS. As a result of these enhanced technological advancements, smaller sample sizes can now be utilized to obtain required method detection limits, reducing solvent and standard consumptions and overall sample preparation time.

This application note describes a reliable, efficient, and cost-effective SPE method utilizing two stacked cartridges for the extraction of 100 mL water samples or less. The upper EC8270 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 compounds with high polarity*, such as 1,4-dioxane, n-nitrosodimethylamine, n-nitrosomethylethylamine, methyl methanesulfonate, ethyl methanesulfonate, and1-Nitrosopyrrolidine. High sample throughout can be achieved by extracting 16 samples simultaneously with a 16-port glass block SPE manifold.

**: A Carbon cartridge is NOT needed if the above mentioned polar analytes are not included in the assay, such as the TCLP SVOC list.*

SPE Procedure:

1. Sample Pretreatment

a) Transfer 100 mL sample, which is dechlorinated with 80 mg/L sodium thiosulfate if free chlorine presents and acidified to pH < 2 with 6 N HCl or H_2SO_4, to a small glass container or use the entire amount that is sampled and preserved in a 100-mL glass bottle.

b) Spike with appropriate levels of surrogates and target analytes for fortified samples.

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

2. SPE Setup

a) Connect the carbon cartridge to the end of the EC8270 cartridge using a cartridge adaptor.

b) Insert a plug of deactivated glass wool into the EC8270 cartridge to prevent sorbent clogging caused by samples with < 1% sediment. Please refer to EPA Method 3535A Section 11.1 for instructions if samples contain > 1% sediment.

c) Attach the large volume sample delivery tube to the top of the EC8270 cartridge, and place the connected SPE cartridges with transfer tubes to an SPE manifold.

Tip 2: The carbon cartridge is not needed if 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) Insert the stainless steel ends of the transfer tubes into a beaker containing dichloromethane (DCM) (10 mL x *n* samples). Apply vacuum for a few seconds so that enough DCM is drawn through the SPE cartridges to wet the sorbents, and let soak for 1 min. Draw the remaining DCM to waste slowly, and leave full vacuum on for 1 min to remove all of the DCM.

b) Insert the stainless steel ends of the transfer tubes into another beaker containing methanol (10 mL x *n* samples), let soak for 1 min, then draw slowly to waste leaving a thin layer above the frit.
c) Repeat step b) with 10 mL of reagent water per sample.

4. Sample Loading

a) Insert the stainless steel ends of the transfer tubes into each corresponding 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 SPE cartridges with 10 mL reagent water.
b) Disconnect the SPE cartridges. Dry both EC8270 and carbon cartridges under full vacuum for 10 min.

Tip 3: Remove as much water as possible. Wet sorbents may result in low recoveries.

6. Analyte Elution

a) Insert the collection rack with 16 x 100 mm test tubes into the manifold.
b) Elute EC8270 and carbon cartridges separately. Apply respective elution solvents to the appropriate SPE cartridge, draw 1/3 through, let soak for 1 min, then draw the remaining through in a slow dropwise fashion. Turn full vacuum on for 1 min after each elution.

EC8270 cartridge	10 mL 1:1 acetone:n-hexane into test tube A (bottle rinse with transfer tube)
	1 cartridge volume of 2% ammonia in DCM into test tube B (Prepared fresh daily)
Carbon cartridge	1 cartridge volume of DCM into test tube C

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

7. Eluate Drying

a) Dry the eluates using a 15-mL fritted reservoir (or a glass funnel stopped with glass wool) holding about 15 g of anhydrous Na_2SO_4 pre-rinsed with 5 mL DCM.
b) Apply the eluates (from test tubes A, C, and B) to the Na_2SO_4 bed and collect in a 60-mL vial.
c) Rinse each individual test tube with 5 mL DCM, and apply the rinses to the Na_2SO_4 bed and collect.

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

8. Concentration

a) Concentrate the dried eluates to 0.7-0.9 mL (or a higher volume depending on the method sensitivity the lab can achieve) using TurboVap (40 ºC water bath with a gentle stream of nitrogen) or other solvent reduction techniques.
b) Add internal standards, transfer the extract to a 2-mL auto-sampler vial, and adjust the final volume to 1 mL with DCM. The samples are ready for instrumental analysis.

Results:

Recovery and RSD in Laboratory Fortified Blanks

(100 mL sample spiked with 100 µg/L of target analytes and surrogates)

Compound Name	Avg Recovery%	RSD% (n=4)
1,4-Dioxane	67.4	6.6
NDMA d6*	85.5	4.6
Pyridine	66.9	7.8
N-nitrosodimethylamine	88.1	4.7
2-Picoline	75.0	7.7
N-nitrosomethylethylamine	88.4	1.6
Methyl methanesulfonate	84.3	1.9
N-nitrosodiethylamine	89.5	1.4
Ethyl methanesulfonate	91.1	0.7
Aniline	64.0	7.9

Phenol d5*	72.6	1.3
Phenol	66.2	4.0
Bis(2-chloroethyl) ether d8*	87.9	1.0
Bis[2-chloroethyl]ether	83.2	3.0
2-Chlorophenol d4*	92.8	1.4
2-Chlorophenol	89.8	0.6
1,3-Dichlorobenzene	66.8	3.5
1,4-Dichlorobenzene	67.3	3.6
Benzyl alcohol	64.1	2.4
1,2-Dichlorobenzene	70.4	3.3
2-Methylphenol	92.1	0.9
Bis[2-chloroisopropyl]ether	85.4	0.5
Acetophenone	91.4	0.8
1-Nitrosopyrrolidine	92.2	0.7
4-Methylphenol d8*	95.0	0.7
3&4-Methylphenol	91.8	0.6
o-Toluidine	62.1	12.8
N-nitro-di-n-propylamine	90.7	1.0
Hexachloroethane	66.8	2.5
Nitrobenzene d5*	91.2	1.9
Nitrobenzene	89.1	1.0
1-Nitrosopiperidine	93.7	0.7
Isophorone	91.6	0.6
2-Nitrophenol d4*	94.2	1.3
2-Nitrophenol	90.7	0.8
2,4-Dimethylphenol	91.8	1.2
Bis[2-chloroethoxy]methane	94.4	1.9
Benzoic acid	142.6	4.9
2,4-Dichlorophenol d3*	94.6	1.2
2,4-Dichlorophenol	91.8	1.7
1,2,4-Trichlorobenzene	72.2	3.0
Naphthalene	78.6	2.3
2,6-Dichlorophenol	90.6	0.1
4-Chloroaniline d4*	59.1	9.7
4-Chloroaniline	57.8	9.6
Hexachloropropene	63.0	2.7
Hexachlorobutadiene	68.6	2.5
N-nitroso di-n-butylamine	91.9	1.2
4-Chloro-3-methylphenol	91.5	2.1
Isosafrole (cis & trans)	84.7	1.9
2-Methylnaphthalene	78.5	2.6

1-Methylnaphthalene	80.4	3.1
1,2,4,5-Tetrachlorobenzene	73.6	3.1
Hexachorocyclopentadiene	74.8	3.9
2,4,6-Trichlorophenol	90.5	1.3
2,4,5-Trichlorophenol	98.1	0.7
Safrole	83.6	1.0
2-Chloronaphthalene	81.2	1.2
1-Chloronaphthalene	78.5	3.9
2-Nitroaniline	95.3	0.7
1,4-Naphthalenedione	84.8	1.2
Dimethylphthalate d6*	97.1	0.8
Dimethyl phthalate	94.0	1.6
Acenaphthylene d8*	85.5	2.6
Acenaphthylene	83.0	1.4
2,6-Dinitrotoluene	93.2	1.0
3-Nitroaniline	84.5	3.1
3-Nitrophenol	94.0	1.7
Acenaphthene	84.7	1.9
2,4-Dinitrophenol	121.8	6.5
Dibenzofuran	85.0	1.8
4-Nitrophenol d4*	96.7	2.1
4-Nitrophenol	79.4	1.5
Pentachlorobenzene	81.2	1.8
2,4-Dinitrotulene	94.2	1.0
1-Naphthalenamine	66.9	6.4
2,3,4,6-Tetrachlorophenol	94.1	1.7
2-Naphthalenamine	148.1	4.0
Diethyl phthalate	95.1	1.7
Fluorene d10*	90.1	1.4
Fluorene	87.1	1.0
4-Chlorophenylphenylether	84.2	1.5
5-Nitro-o-toluidine	84.8	3.2
4-Nitroaniline	96.2	2.5
4,6-Dintro-2-methylphenol d2*	106.9	4.4
4,6-Dinitro-2-methylphenol	104.9	4.1
Diphenylamine	92.1	1.1
Azobenzene	89.3	1.7
Diallate (cis & trans)	92.9	1.1
1,3,5-Trinitrobenzne	92.4	1.7
Bromophenoxybenzene	86.6	1.7
Phenacetin	101.8	1.2

Hexachlorobenzene	87.5	1.1
4-Aminobiphenyl	82.5	4.8
Pentachlorophenol	96.6	1.5
Pronamide	98.5	0.2
Pentachloronitrobenzene	91.7	1.6
Phenanthrene	90.0	1.9
Dinoseb	105.5	2.2
Anthracene d10*	91.8	1.6
Anthracene	89.3	2.0
Dibutyl phthalate	99.6	0.8
Isodrin	88.0	1.5
Fluoranthene	94.4	1.3
Benzidine	31.0	16.1
Pyrene d10*	97.9	0.9
Pyrene	94.4	1.3
p-Dimethylaminoazobenzene	85.0	2.7
Chlorobenzilate	97.5	0.6
Benzyl butyl phthalate	98.6	0.9
3,3'-Dimethylbenzidine	42.2	11.4
2-Acetylaminofluorene	102.4	1.3
Benz[a]anthracene	97.0	1.3
3,3'-Dichlorobenzidine	66.9	3.4
Chrysene	90.7	0.7
Bis(2-ethylhexyl) phthalate	93.9	1.3
Di-n-octyl phthalate	93.7	1.2
Benzo[b]fluoranthene	91.4	1.1
Benzo[k]fluoranthene	91.5	0.3
Benzo(a)pyrene d12*	93.6	1.1
Benzo[a]pyrene	89.3	1.9
3-Methylcholanthrene	89.6	1.9
Indeno[123-cd]pyrene	93.0	0.7
Dibenz[ah]anthracene	93.2	1.2
Benzo[ghi]perylene	91.2	1.4
Overall mean	87.2	2.5

* Denotes surrogates used during analysis

Note: For the extraction of samples with 100 to 1000 mL volumes, please refer to: https://www.unitedchem.com/sites/default/files/docs/spapplications/5108-04-01-uct8270_08102015_updated.pdf

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