Implementation of EPA Method 8270E Using Solid-Phase Extraction and Hydrogen as Carrier Gas for GC/MS Analysis

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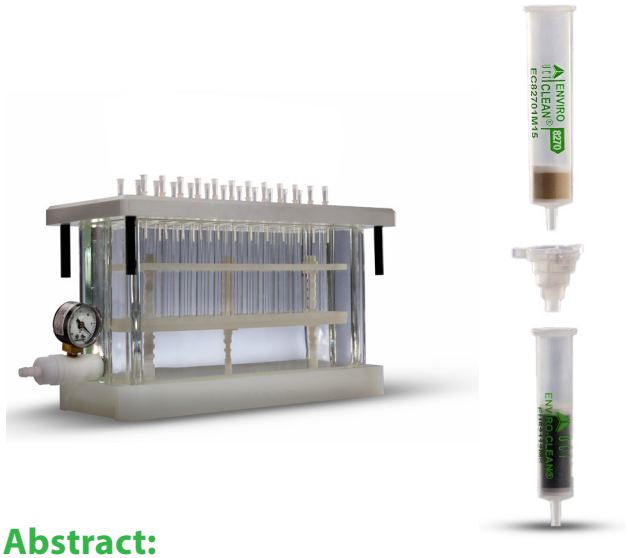

UCT Featured Products

EC82702M15
2000 mg 8270 sorbent
15 mL cartridge

ECSS25K
Enviro-Clean® Bulk Sodium Sulfate Anhydrous, ACS grade, granular, 60 mesh

VMF016GL
Glass Block Vacuum Manifold System - 16 position

VMF02125
Glass Manifold Polypropylene Collection Rack VOA Vials - 12 Position System

VMFSPEVAPCR-3252
27-29mm, VOA Vial Collection Tray 32 Position

EU52113M6
3000 mg activated carbon
6 mL cartridge*

VMFSTFR12
Large volume sample transfer tubes

AD0000AS
Cartridge adaptor

RFV1F25P
Fritted Reservoir 1 Frit, 10 Micron 25 mL

RFT01F8P
Fritted Reservoir 1 Frit, 20 Micron 6 mL

VMFSPEVAP-32
32 Position SPeVAP module w/ install kit

**For very polar analytes only (1,4-dioxane, n-nitrosodimethylamine, n-nitrosomethylethylamine, methylmethanesulfonate, ethyl methanesulfonate, and 1-nitrosopyrrolidine)*

Abstract:

This application note describes an extraction method for isolating Semivolatile Organic Compounds (SVOCs) in accordance with EPA Method 8270E. A proprietary 8270 sorbent was used to selectively isolate acidic, basic, and neutral compounds. Combined with an activated carbon cartridge, this setup enhances the retention of polar analytes during solid-phase extraction (SPE) before GC/MS analysis. High sample throughput is achieved by extracting 12 samples simultaneously with a 16-port glass block SPE manifold.

Introduction:

EPA Method 8270E is the latest update to the standardized protocol for analyzing semi-volatile organic compounds (SVOCs) in water and other matrices. It employs gas chromatography/mass spectrometry (GC/MS) to identify and quantify a broad range of compounds, encompassing acidic, basic, and neutral analytes. The method allows the use of SPE as a sample preparation option. Compared to traditional liquid/liquid and separatory funnel extractions, SPE drastically reduces solvent use and streamlines workflows, making the method more environmentally friendly and cost-effective. These advancements ensure compliance with regulatory standards while maintaining robust analytical performance.

The SPE procedure for EPA 8270 requires optimization of operating parameters and the careful selection of elution solvents, due to the wide range of compounds with varying physicochemical properties. These compounds include nonpolar, moderately polar, and highly polar analytes, which utilize different mechanisms of SPE working in tandem to capture them all sufficiently. UCT's proprietary 8270 sorbent is engineered to selectively isolate acidic, basic, and neutral compounds, while the incorporation of an activated carbon cartridge facilitates the retention of highly polar analytes, such as 1,4-dioxane.

UCT has demonstrated that this method is capable of detecting 133 analytes at concentration levels of 10 µg/L or less, including semivolatile compounds from the EPA Priority Pollutant List (PPL), the RCRA Appendix IX list, and the Superfund Analytical Methods target list (SFAM01.1). The method achieves high sample throughput by extracting multiple samples simultaneously with a multiport SPE glass block manifold. GC/MS analysis was conducted using hydrogen as the carrier gas, reflecting the industry's shift from helium to a more cost-effective and sustainable alternative.

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Experimental

a) Sample Preparation Procedure

SPE Format

Enviro-Clean® 8270 Extraction Cartridges 2000 mg 15 mL (EC82702M15) and Enviro-Clean® 521 3000 mg Activated Carbon 6 mL (EU52113M6).

Sample Pre-treatment

a) Dechlorinate the 1-liter sample with 80 mg/L of sodium thio sulfate if free chlorine is present.
b) Adjust sample pH to < 2 using 6N HCl or H_2SO_4. (Check LCS and MB pH with a meter).
c) Spike with surrogates and target analytes for fortified samples.

Tip: *Prepare spiking solutions in water-miscible solvents to avoid degradation of the analytes during storage. Care should be taken, as some analytes will interact with one another when combined, therefore multiple spiking solutions (and possibly calibration standards) may need to be prepared. For more information, see 8270E section 1.4.14. Check with your reference material provider.*

SPE System Setup

a) Connect the carbon cartridge to the end of the 8270-cartridge using a cartridge adaptor (AD0000AS).
b) Insert a loose plug of deactivated glass wool into the 8270 cartridges to prevent the sorbent from clogging when samples contain a high particulate content. 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.
d) Attach the connected SPE cartridges to the SPE manifold (VMF016GL).

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).
c) Draw the entire sample through the cartridges.

Cartridge Conditioning

a) Sorbent rinsing - Insert the stainless-steel ends of the transfer tubes into a beaker containing dichloromethane (DCM) (15 mL per sample).
b) Apply vacuum for a few seconds to draw enough DCM through the SPE cartridges to rinse the sorbents (approximately 10 mL).
c) Allow the sorbent to soak in DCM for 1 min.
d) Slowly draw the remaining DCM to waste.
e) Apply full vacuum for 3 minutes to remove all the DCM from the sorbent.
f) Sorbent conditioning - Insert the stainless-steel ends of the transfer tubes into a beaker containing methanol (15 mL per sample).
g) Apply vacuum for a few seconds to draw enough methanol through the SPE cartridges to wet the sorbents (approximately 10 mL).
h) Slowly draw off the excess methanol to waste, leaving a thin layer above the frit.
i) Sorbent equilibration – Insert the stainless-steel ends of the transfer tubes into a beaker containing 0.05N HCl in water (15 mL per sample).
j) Apply vacuum for a few seconds to draw enough 0.05N HCl in water through the SPE cartridges to equilibrate the sorbents (approximately 10 mL).
k) Slowly draw off the excess water to waste, leaving a layer above the frit.

Rinse and Dry Cartridges

a) Rinse the sample bottle with 10 mL of deionized water. Allow water to pass through the SPE cartridge until dryness
b) Disassemble the 8270 SPE cartridge from the carbon cartridge and remove the adapter.
c) Place the 8270 cartridges with transfer tube and the carbon cartridges on separate positions on the glass block manifold.
d) Dry the 8270 cartridges under full vacuum for 10 minutes, and the carbon cartridges for 15 minutes.

Tip: *Remove as much water as possible. Wet sorbents will result in low recoveries. Full vacuum pressure should equal 25" Hg (635 mm Hg).*

Analyte Elution

a) Insert the collection rack (VMF02125) with 40-60 mL glass vials into the manifold.
b) Elute the 8270 and carbon cartridges separately. There will be separate elutions of the 8270 cartridges into 2 vials (Collection vial A and B).

Elution 1: EC8270 Cartridge - Collection Vial A

1. Place the EC8270 cartridge over Collection Vial A.
2. Add 10 mL of 1:9 acetone: n-hexane to the bottle and rinse it well.
3. Draw the solvent from the bottle into the EC8270 cartridge and let it soak for 2 minutes.
4. Dropwise, draw the solvent into Collection Vial A.
5. Turn this cartridge on full vacuum for 1 minute.

Tip: *The bottle rinse is critical for good recovery of PAHs which stick to glass. If recoveries of phenols or benzidine are not of concern, a higher concentration of acetone can be used for faster evaporation time.*
(i.e. 1:1 acetone:n-hexane)

Elution 2: EC8270 Cartridge - Collection Vial B

1. Place the EC8270 cartridge over Collection Vial B and add 10 mL of 18:2:80 IPA:NH_4OH:DCM to the cartridge.

Note: *This solvent must be made fresh daily, with NH_4OH less than 6 months old. Add ammonium hydroxide to IPA, stir, and then combine with DCM to ensure miscibility.*

2. Allow the solvent to soak for 2 minutes.
3. Dropwise, draw the solvent into Collection Vial B.
4. Turn this cartridge on full vacuum for 1 minute.
5. Repeat step (2) with 15 mL of DCM.

Elution 3: Carbon Cartridge

1. Add 15 mL of DCM to the carbon cartridge

Optional: *to remove excess water from the carbon cartridge, a 6 mL fritted reservoir filled with 3 g of DCM rinsed sodium sulfate can be attached in line with the carbon cartridge. This should not be done in place of the drying step, but as an additional measure.*

2. Allow the solvent to soak for 2 minutes.
3. Dropwise, draw the solvent into its collection vial.
4. Dry this cartridge on full vacuum for 1 minute.

Eluate Drying with Sodium Sulfate Anhydrous

a) Fill a 25 mL fritted reservoir with 15 to 20 g of Na_2SO_4 for each sample. Alternatively, use product ECUNIMSS (80 mL cartridge containing 20 g muffled sodium sulfate).

b) Rinse the sodium sulfate with DCM and discard the rinsate.

c) Place a 60 mL vial in the manifold to capture the dried eluate.

d) Pour each of the 3 eluents through the sodium sulfate and collect.

Tip: *If sodium sulfate starts to clump, agitate it with a glass stirring rod.*

e) Rinse each vial with 2 x 5 mL of DCM and transfer it to sodium sulfate. If the sodium sulfate is discolored, continue rinsing with DCM until it is white. Breaking up clumps in the sodium sulfate with a stir rod may be necessary in this case.

Concentration

a) Concentrate the eluates to 8-10 mL at 40-45 °C, either using SPeVAP or equivalent with a gentle stream of nitrogen, or a water bath and K-D apparatus.

b) Transfer eluate to a class-A graduated 25 mL jacketed concentrator tube, rinsing the vial twice with approximately 2 mL DCM to ensure full transfer of the sample.

c) Gently concentrate to approx. 0.6 - 0.8 mL at 35 °C using a gentle stream of nitrogen on an Organomation N-EVAP or equivalent, adjusting the needle position so a small 'dimple' is formed on the surface.

d) Remove from the water bath and rinse the sides of the concentrator tube with approx. 100 µL of DCM

e) With a Pasteur pipette, mix the extract, rinsing the sides of the concentrator tube.

f) Transfer the extract to a GC vial and bring to a 1 mL final volume with DCM.

g) Add internal standard.

h) Analyze by GC/MS.

Note: *Concentration should occur immediately after the eluate drying step to ensure recovery for light sensitive compounds.*

b) Analytical Conditions

The GC/MS method is summarized in **Table 1.**

Table 1. GC/MS Conditions

GC/MS	Agilent 6890/5975C
Column	Rxi-5Sil MS, 30m x 250 µm x 0.25 µm
Inlet Temp.	280 °C
Injection	1 µL; Split 10:1
Liner	Restek Topaz Splitless, 4mm
Oven	40°C (1 min) to 280 °C at 20 °C/min, 300 °C at 5 °C /min
Carrier gas	Hydrogen at 1.2 mL/min constant flow rate
MS Parameters	Source at 230°C; Quad at 150 °C; transfer line temp. at 280 °C; Electron energy at 70 eV
Detector	MSD Quadrupole, full scan mode (EI), 50-500 amu
Tune File	DFTPP.U

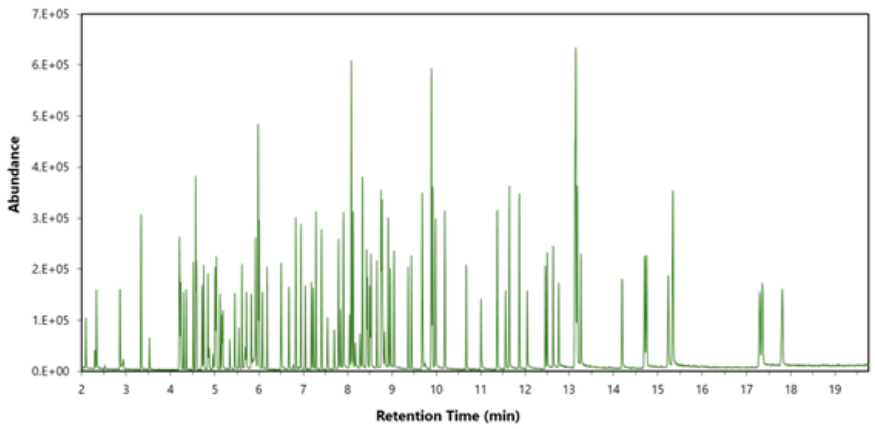

Figure 1. Chromatogram at 20 mg/L of the continuing calibration SVOCs standard mixture A (97 compounds), 8270 megamix, basic/neutral surrogates, methapyriline, benzoic acid, benzidine, 3,3'-dichlorobenzidine, appendix IX mix 1, acid surrogates (40 mg/L), and internal standards (40 mg/L).

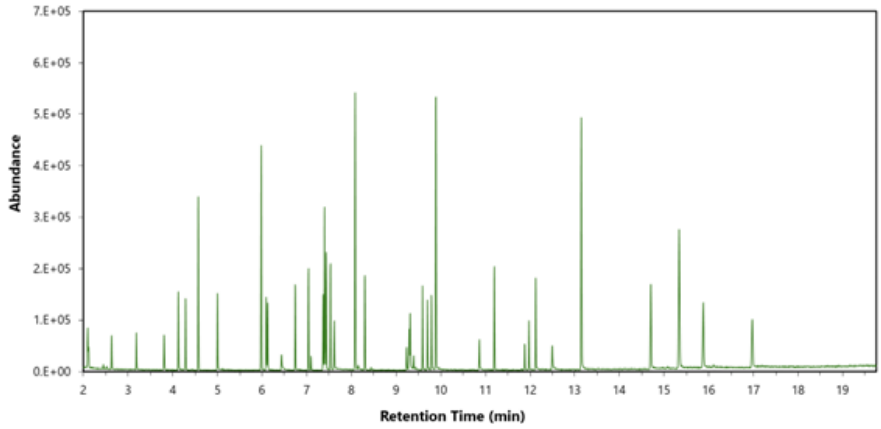

Figure 2. Chromatogram at 20 mg/L of the continuing calibration SVOCs standard mixture B (33 compounds), Appendix IX mix 2, and internal standards (40 mg/L).

Results and Discussion:

Initial demonstration of proficiency (IDP) was performed in fortified samples (133 compounds) at different concentration levels (1 µg/L, 5 µg/L and 20 µg/L). Limits of detection (LOD) were calculated and are summarized in Tables 2 and 3. Chromatograms of the fortified samples and blanks are shown in Figures 1-3. Fortified water samples (n=7) at a concentration of 1 µg/L were analyzed over a three-day period to determine the LOD for the analytes. The results are summarized in Table 3. At a lower limit of quantification (LLOQ) of 1 µg/L, 81 out of the 133 compounds showed acceptable recovery (49-121%) and precision (4-20%) values. However, exceptions were observed for 1,4-naphthoquinone (RSD 42%) and 2,4-dinitrophenol (RSD 26%).

Samples spiked at 5 µg/L (10 µg/L for benzidine, 3,3'-dichlorobenzidine, methapyrilene, and benzoic acid) were analyzed over three days to assess the LOD for the remaining 52 analytes of interest. Table 2 shows that the recovery values for these analytes ranged from 51% to 122%, with RSD values between 1% and 17%. However, benzoic acid exhibited a recovery value within the acceptable range but an RSD value of 27%. This specific compound is known to present challenges when extracted from water matrices. Additionally, 1,4-phenylenediamine failed to achieve sufficient recovery during the LLOQ studies

Finally, an IDP was conducted for all compounds by extracting four replicates near the midpoint of the calibration curve, at 20 µg/L. The recoveries for these samples were evaluated and are reported in Table 2. At this concentration, recovery values ranged from 56% to 121%, with RSD values ≤20%. The compounds 1,4-phenylenediamine (34%) and hexachloropropene (41%) did not meet acceptable recovery criteria. These compounds are either known to pose challenges in extraction methods or lack historical data for aqueous extraction techniques.

Table 2. Quality Control Data.

Analyte	IDP/LLOQ 5 µg/L*(n=4)		IDP 20 µg/L (n=4)		LOD µg/L (n=7)
	Average Recovery (%)	RSD (%)	Average Recovery (%)	RSD (%)	
1,4-Dioxane	73	8	61	16	1.28
N-Nitrosodimethylamine	89	4	78	14	1.02
Pyridine	85	4	73	13	1.12
Ethyl Methacrylate	87	7	70	14	1.19
2-Picoline	101	4	84	11	1.09
N-Nitrosomethylethyl-amine	100	12	88	11	1.91
Methyl Methanesulfonate	96	8	84	12	0.65
2-Fluorophenol	95	5	86	13	N/A (Surr)
N-Nitrosodiethylamine	86	4	85	15	1.09
Ethyl Methanesulfonate	95	10	81	13	1.50
Benzaldehyde	100	3	83	13	0.98
Phenol-d6	94	5	80	12	N/A (Surr)
Phenol	93	6	81	11	0.91
Aniline	65	4	68	12	0.55
Bis(2-chloroethyl) ether	91	8	79	14	0.99
Pentachloroethane	86	13	73	15	1.10
2-Chlorophenol	100	7	89	14	1/09
Acetophenone	105	4	94	9	0.97
1,3-Dichlorobenzene	83	2	70	16	0.62
1,4-Dichlorobenzene	80	4	71	16	0.31
Benzyl Alcohol	122	6	91	11	2.47
1,2-Dichlorobenzene	86	5	76	14	0.56
2-Methylphenol	96	5	89	11	0.90
2,2'-oxybis(1-chloropro-pane)	91	7	79	13	0.52
N-Nitrosopyrrolidine	81	3	83	12	0.70
3 and 4-Methylphenol	96	6	92	9	1.02
N-Nitrosodi-N-propyl-amine	87	2	97	11	1.30
4-Nitrosomorpholine	81	10	92	10	1.28
o-Toluidine	85	4	82	9	0.65
Hexachloroethane	87	1	76	19	0.77
Nitrobenzene-d5	89	8	84	11	N/A (Surr)
Nitrobenzene	95	5	84	13	1.31
N-Nitrosopiperidine	94	10	89	6	1.22
Isophorone	83	6	86	8	0.69
2-Nitrophenol	86	4	87	11	0.82
2,4-Dimethylphenol	90	5	91	8	0.60
Benzoic Acid*	80	27	71	12	6.28
Bis(2-chloroethoxy) methane	87	6	81	10	0.81
2,4-Dichlorophenol	96	10	99	9	1.28
1,2,4-Trichlorobenzene	88	5	81	12	0.53
Phentermine	103	5	89	3	1.36
Naphthalene	93	4	82	11	0.49
4-Chloroaniline	76	8	82	8	0.81
2,6-Dichlorophenol	90	2	93	8	0.52
Hexachloropropene	51	5	41	15	0.54
Hexachlorobutadiene	84	4	78	15	0.62
Caprolactam	105	8	90	6	2.24
N-Nitrosodi-n-butylamine	99	4	90	6	1.40
1,4-Phenylenediamine**	0	0	34	5	**
4-Chloro-3-methylphenol	94	6	95	4	0.77
Isosafrole I	88	6	88	8	0.72
2-Methylnaphthalene	95	4	85	9	0.59
1-Methylnaphthalene	91	4	84	9	0.44
Hexachlorocyclopenta-diene	87	4	80	13	0.77
1,2,4,5-Tetrachloroben-zene	86	4	82	11	0.65
Isosafrole II	91	6	92	7	1.65
2,4,6-Trichlorophenol	97	4	100	8	1.09
2,4,5-Trichlorophenol	96	4	99	6	0.68
2-Chloronaphthalene	94	2	90	11	0.35
1-Chloronaphthalene	92	6	85	11	0.65

Table 2. *(continued)*

Analyte	IDP/LLOQ 5 µg/L* (n=4)		IDP 20 µg/L (n=4)		LOD µg/L (n=7)
	Average Recovery (%)	RSD (%)	Average Recovery (%)	RSD (%)	
2-Fluorobiphenyl	87	2	85	8	N/A (Surr)
Safrole	85	4	90	9	0.88
1,1'-Biphenyl	93	4	89	9	0.55
Diphenyl Ether	88	2	91	8	0.61
2-Nitroaniline	82	3	94	6	1.16
1,4-Naphthoquinone	56	6	75	8	0.22
1,4-Dinitrobenzene	84	6	90	5	1.25
Dimethylphthalate	90	6	97	7	0.60
1,3-Dinitrobenzene	91	9	92	5	0.82
2,6-Dinitrotoluene	88	4	95	7	0.79
1,2-Dinitrobenzene	85	6	93	7	0.26
Acenapthylene	89	2	91	8	0.33
3-Nitroaniline	71	11	83	7	0.78
Acenaphthene	95	4	89	8	0.42
2,4-Dinitrophenol	98	14	76	7	1.92
4-Nitrophenol	93	17	95	6	2.05
Pentachlorobenzene	88	6	89	8	0.92
2,4-Dinitrotoluene	79	6	92	6	0.67
Dibenzofuran	94	5	91	7	0.47
1-Naphthylamine	62	8	87	6	1.04
2,3,5,6-Tetrachlorophenol	94	3	101	5	0.53
2,3,4,6-Tetrachlorophenol	99	6	100	4	0.71
2-Napthtylamine	64	6	84	5	0.63
Diethylphthalate	64	5	83	4	0.52
4-Chlorophenyl phenyl ether	90	3	95	8	0.55
Fluorene	92	3	91	6	0.41
4-Nitroaniline	81	9	93	6	1.29
4,6-Dinitro-2-methyl-phenol	112	4	91	2	0.94
N-Nitrosodiphenylamine	88	4	97	5	0.30
Diphenylhydrazine	80	5	93	6	0.56
2,4,6-Tribromophenol	106	8	109	5	N/A (Surr)
1,3,5-Trinitrobenzene	119	5	83	4	0.97
Phenacetin	113	4	103	3	0.66
Diallate	85	3	99	6	1.00
4-Bromophenyl phenyl ether	93	3	96	6	0.41
Hexachlorobenzene	93	7	99	6	0.68
Atrazine	117	4	89	2	0.78
Pentachlorophenol	109	7	114	3	1.11
4-Aminobiphenyl	62	7	81	3	0.91
Pentachloronitrobenzene	93	7	100	4	1.30
Phenanthrene	94	3	94	5	0.34
Propyzamide	79	10	97	5	0.89
Anthracene	84	5	93	5	0.48
Carbazole	89	5	97	3	0.47
Di-n-butylphthalate	112	5	100	2	3.25
4-Nitroquinoline-1-oxide	62	12	90	3	1.97
Methapyrilene*	70	8	86	4	2.48
Isodrin	93	6	101	5	1.21
Fluoranthene	90	3	97	4	0.25
Benzidine*	38	4	58	7	1.53
Pyrene	95	3	100	7	0.48
Aramite I	102	14	97	7	0.99
o-Terphenyl-D14	97	3	98	4	N/A (Surr)
Aramite II	84	10	85	5	1.02
p-Dimethylaminoazo-benzene	105	4	111	4	0.93
3'3-Dimethylbenzidine*	57	5	121	6	0.86
Butylbenzylphthalate	108	4	97	5	1.08
Kepone	92	16	97	4	1.30
Bis(2-ethylhexyl)adipate	88	6	84	7	0.85
2-Acetylaminofluorene	76	2	95	4	0.44
3'3-Dichlorobenzidine	67	7	95	3	1.38
Benz[a]anthracene	84	4	97	6	0.18

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Table 2. *(continued)*

Analyte	IDP/LLOQ 5 µg/L* (n=4)		IDP 20 µg/L (n=4)		LOD µg/L (n=7)
	Average Recovery (%)	RSD (%)	Average Recovery (%)	RSD (%)	
Chrysene	84	2	95	6	0.40
Bis(2-ethylhexyl)phthalate	89	5	97	5	2.47
Di-n-octylphthalate	101	2	94	4	0.56
7,12-Dimethylbenzo[a]anthracene	87	4	93	5	0.78
Benzo[b]fluoranthene	95	4	106	4	0.53
Benzo[k]fluoranthene	87	4	95	7	0.67
Benzo[a]pyrene	86	2	91	6	0.61
3-Methylchloanthrene	84	5	89	6	0.40
Dibenz[a,j]acridine	52	4	56	9	0.86
Indeno[1,2,3-cd]pyrene	91	5	93	7	0.66
Dibenz[a,h]anthracene	89	6	91	6	0.57
Benzo[g,h,i]perylene	91	4	91	6	0.70

**Compounds fortified at 10 µg/L*
***1,4-Phenylenediamine could not be recovered at LLOQ*

Table 3. LOD and LOQ Study Results at 1 µg/L.

Analyte	LLOQ 1 µg/L (n=7)		LOD µg/L
	Average Recovery (%)	RSD (%)	
N-Nitrosodimethylamine	81	15	0.40
Pyridine	71	12	0.30
Ethyl Methacrylate	80	9	0.23
2-Picoline	92	12	0.38
N-Nitrosomethylethylamine	106	11	0.40
Methyl Methanesulfonate	88	13	0.37
2-Fluorophenol	88	8	N/A (Surr)
N-Nitrosodiethylamine	84	7	0.21
Ethyl Methanesulfonate	100	20	0.68
Benzaldehyde	121	11	0.67
Phenol-d6	87	9	N/A (Surr)
Phenol	87	11	0.32
Bis(2-chloroethyl) ether	82	10	0.27
2-Chlorophenol	81	9	0.24
1,3-Dichlorobenzene	72	7	0.17
1,4-Dichlorobenzene	72	9	0.22
1,2-Dichlorobenzene	76	6	0.16
2-Methylphenol	85	14	0.41
2,2'-oxybis(1-chloropropane)	83	14	0.40
Hexachloroethane	79	7	0.18
Nitrobenzene-d5	74	9	N/A (Surr)
Nitrobenzene	81	10	0.26
Isophorone	81	8	0.22
2-Nitrophenol	77	9	0.24
Bis(2-chloroethoxy)methane	83	6	0.18
2,4-Dichlorophenol	70	11	0.25
1,2,4-Trichlorobenzene	77	10	0.27
Naphthalene	81	7	0.19
2,6-Dichlorophenol	73	12	0.30
Hexachlorobutadiene	70	6	0.13
N-Nitrosodi-n-butylamine	85	20	0.57
4-Chloro-3-methylphenol	89	4	0.13
Isosafrole I	77	7	0.19
2-Methylnaphthalene	88	6	0.28
1-Methylnaphthalene	80	5	0.15

Table 3. *(continued)*

Analyte	LLOQ 1 µg/L (n=7)		LOD µg/L
	Average Recovery (%)	RSD (%)	
Hexachlorocyclopentadiene	72	10	0.60
1,2,4,5-Tetrachlorobenzene	83	7	0.20
2,4,6-Trichlorophenol	76	17	0.43
2,4,5-Trichlorophenol	85	5	0.13
2-Chloronaphthalene	80	8	0.21
1-Chloronaphthalene	79	8	0.21
2-Fluorobiphenyl	81	7	N/A (Surr)
Safrole	71	6	0.15
1,1'-Biphenyl	85	8	0.24
Diphenyl Ether	78	5	0.15
2-Nitroaniline	67	10	0.23
1,4-Naphthoquinone	49	42	0.70
Dimethylphthalate	69	10	0.23
1,3-Dinitrobenzene	73	8	0.21
2,6-Dinitrotoluene	70	9	0.21
1,2-Dinitrobenzene	72	15	0.37
Acenapthylene	75	9	0.22
Acenaphthene	89	7	0.21
2,4-Dinitrophenol	78	26	0.68
Pentachlorobenzene	75	7	0.17
Dibenzofuran	80	6	0.15
2,3,4,6-Tetrachlorophenol	75	20	0.50
4-Chlorophenyl phenyl ether	71	6	0.15
Fluorene	83	8	0.23
N-Nitrosodiphenylamine	82	7	0.19
Diphenylhydrazine	76	6	0.17
2,4,6-Tribromophenol	87	11	N/A (Surr)
Diallate	89	17	0.52
4-Bromophenyl phenyl ether	84	10	0.27
Hexachlorobenzene	89	12	0.36
Pentachlorophenol	90	17	0.53
Pentachloronitrobenzene	80	16	0.44
Phenanthrene	89	6	0.18
Anthracene	78	7	0.19
Carbazole	79	6	0.17
Isodrin	80	13	0.36
Fluoranthene	82	18	0.50
Pyrene	99	15	0.52
o-Terphenyl-D14	98	5	N/A (Surr)
Aramite II	103	19	0.67
Butylbenzylphthalate	94	16	0.53
Benz[a]anthracene	90	14	0.44
Chrysene	98	19	0.62
Di-n-octylphthalate	83	16	0.46
Benzo[b]fluoranthene	88	15	0.46
Benzo[k]fluoranthene	91	19	0.59
Benzo[a]pyrene	81	13	0.37
3-Methylchloanthrene	73	8	0.19
Dibenz[a,j]acridine	76	17	0.43
Indeno[1,2,3-cd]pyrene	88	16	0.47
Dibenz[a,h]anthracene	90	18	0.54
Benzo[g,h,i]perylene	92	16	0.50

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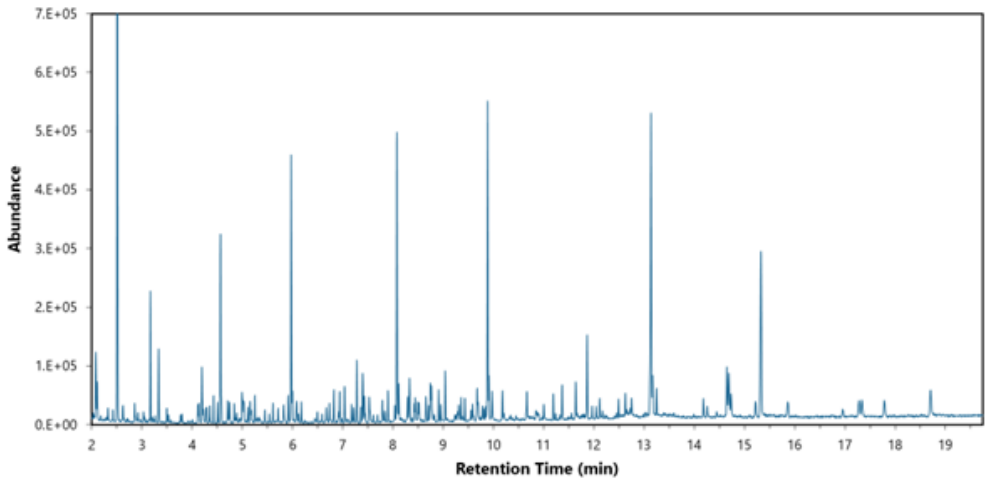

Figure 3. Total Ion Chromatogram (TIC) of the 1L fortified sample at 5 and 10 µg/L (with internal standards at 40 µg/L). (133 Compounds).

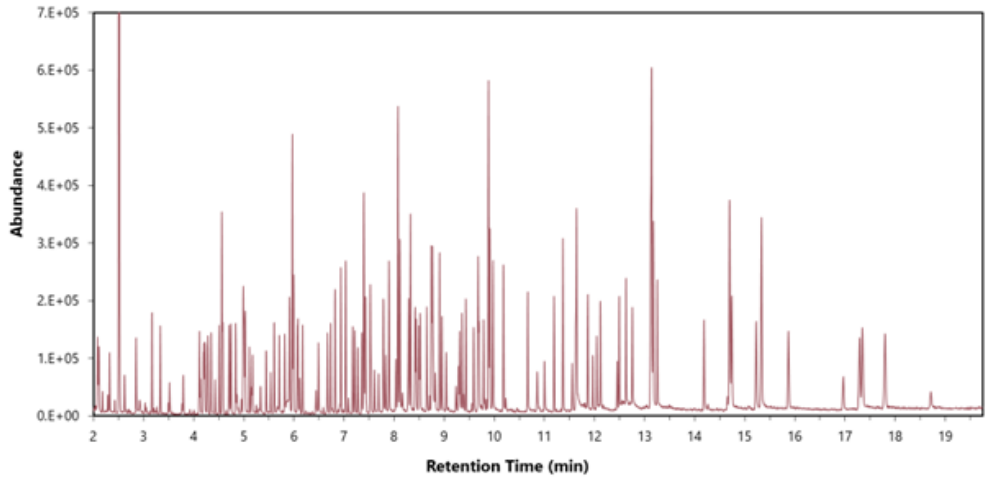

Figure 4. Total Ion Chromatogram (TIC) of the 1L fortified sample at 20 µg/L (with internal standards at 40 µg/L). (133 Compounds).

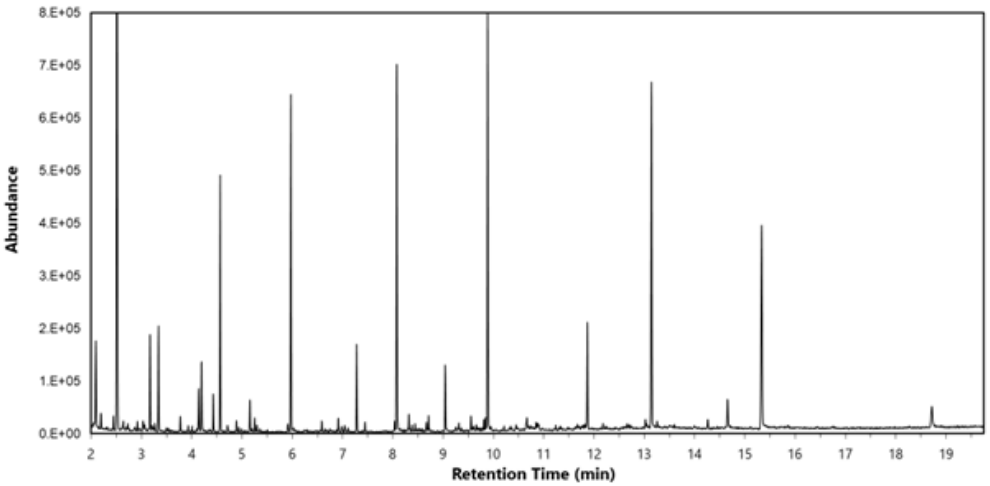

Figure 5. Total Ion Chromatogram (TIC) of the method blank with surrogates at 10 and 20 µg/L and internal standard at 40 µg/L.

Conclusion:

This article outlines the workflow for EPA Method 8270E, employing solid-phase extraction in conjunction with GC/MS analysis using hydrogen as the carrier gas. According to EPA Method 8270E, the recovery of laboratory-spiked blank water samples is considered valid for an extraction method if recoveries fall within the range of 50-150% (laboratories shall develop their own in-house recovery limits after collecting sufficient data per method 8000D section 9.6), with reproducibility not exceeding 20%. The results demonstrate that acceptable accuracy and precision were achieved for the analytes studied across various concentration levels, using EC8270 and EU521 SPE cartridges, along with the VMF016GL Glass Block Vacuum Manifold System, in combination with GC/MS analysis using hydrogen as the carrier gas.

Therefore, this SPE method for extracting a wide variety of acidic, basic, and neutral semivolatile compounds offers laboratories the ability to improve efficiency by increasing sample throughput while minimizing solvent waste and exposure. Moreover, this approach delivers analytical results that are both consistent and comparable to those obtained using traditional aqueous extraction methods.

References:

[1] U.S. Environmental Protection Agency. Method 8270E: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). EPA-600/R-13/469, 2013.

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