Improving Automated SPE Disk Extraction Technology for Semivolatile Compounds in Groundwater Using US EPA Method 8270E

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Key Words: *Groundwater, US EPA Method 8270E, SVOC, SPE, Solid Phase Extraction*

Introduction

Semivolatile organic compounds (SVOC) have a variety of chemical properties that have been found to cause harmful effects to both humans and the environment. Accurate measurements are challenging to obtain because SVOCs readily adsorb onto surfaces, and are found in common household items such as cleaning agents, personal care products, electrical components, pesticides, water and food. Effects to health depend on the chemical nature of the compound in conjunction with the degree of exposure, yet have been known to include allergenic symptoms, reproductive and endocrine issues. Laboratories around the world measure these compounds in water, soil, and leachates from waste sites. US EPA Method 8270E can be used to determine the concentration of SVOCs extracted from liquid, solid and leachate samples in effort to limit exposure and the spread of these persistent organic pollutants. [1]

While almost all laboratories test for fewer than the full list of 243 compounds included in the method, typical laboratories will often measure a large suite of 80 to 100 compounds. Compound classes that can be extracted using this method include: polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols.

This application note will demonstrate the results of Initial Demonstration of Proficiency (IDP) evaluations for compliance with US EPA Method 8270E to determine a list of 114 semi-volatile organic compounds that are neutral, acidic, and basic. In addition to the IDP, a typical groundwater sample will be evaluated and contains a different matrix and particulate content than seen in the IDP. The Matrix Spike (MS)/ Matrix Spike Duplicate (MSD) will demonstrated. Solid phase extraction is described as a suitable sample preparation alternative in Method 8270 and companion method US EPA 3535 outlines the general use of SPE. Suitable sorbent material in disk format for this list of analytes and a modern system for automation will be demonstrated.[2]

Experimental

Outline of SPE Procedure/Experimental

The SVOCs in this method were extracted using automated sample preparation solutions for solid phase extraction (SPE), drying and concentration. The samples were extracted using an Atlantic® One Pass Disk (Biotage) which is a mixed mode disk containing several functionalities. Two different pre filters, Atlantic Fast Flow 1 µm and 5 µm (Biotage) with a fine mesh screen were used, as well as a Carbon Cartridge (Biotage) to ensure adequate retention of difficult to obtain light end compounds, such as NDMA and N-Nitrosomethyl ethylamine.

The Fast Flow Disk Holder was used with the samples because groundwater and leachate samples can at times, have varying levels of particulate matter. The Fast Flow Disk Holder uses a 47 mm disk, but allows larger diameter pre-filters to be placed on top to shield the SPE disk from particulates that may cause clogging, and maintain fast sample flow through the disk. The particulates are retained on the filters and washed with solvent during the elution steps, so any compounds that have been adsorbed on the particulates will be included in the extract.

The extraction was completed using the Biotage® Horizon 5000 (previously known as the SPE-DEX® 5000) (Figure 1); an automated extractor which allows various types of liquid samples to be processed directly from the original sample container. The system includes intuitive software for easy programming, and automatic sample and solvent delivery.

The one liter samples were spiked and acidified to pH2 with hydrochloric acid (HCl) and placed on the Biotage® Horizon 5000. Each of the three positions on the Biotage® Horizon 5000 was set up with the One Pass Kit (Biotage). The resulting extracts were dried using DryDisk® Membranes (Biotage) and concentrated with a DryVap® (Biotage) (Figure 2) for automatic in-line drying and concentration.

The extraction method used with the Biotage® Horizon 5000 is shown in Table 1. The conditions for operating the DryVap® are shown in Table 2. Most of the steps using the Biotage® Horizon 5000 were completely automated and require no operator intervention. For the few steps where a new flask is required for an elution step, a clear message is shown in the software to make the operation of the system simple to follow.

Figure 1. Biotage® Horizon 5000 (previously known as the SPE-DEX 5000) Extractor with One-pass System.

Figure 2. DryVap® System.

Table 1. Biotage® Horizon 5000 Extraction Method.

Step	Operation	Message	Attachment
1	Pause with Message	Part 1 of 3: Neutrals and Acids Elution. Have the Fast Flow Disk Holder with One Pass disk, 1 µm filter, 5 µm filter, top screen over the filters, 250 mL collection flask, and carbon cartridge installed. The down spout of the water in valve must push down on the top screen. Click “Continue” to start Part 1.	None

Step	Operation	Solvent	Solvent Vol. (mL)	Purge Time (s)	Pump Rate (#)	Sat. Time (s)	Soak Time (s)	Drain Time (s)
2	Condition SPE Disk	Acetone	40	60	4	2	60	60

Step	Operation	Sample Flow Rate (#)	Done Loading Sample Delay (s)
4	Load Sample	5	45

Step	Operation	Solvent	Solvent Vol. (mL)	Purge Time (s)	Pump Rate (#)	N2 Blanket	Sat. Time (s)	Soak Time (s)	Drain Time (s)
5	Wash Sample Container	Reagent Water	20	30	4	Off	2	5	30

Step	Operation	Dry Time (s)	Pump Rate (#)	N2 Blanket
6	Air Dry Disk Timer	360	6	Off

Step	Operation	Solvent	Solvent Vol. (mL)	Purge Time (s)	Pump Rate (#)	N2 Blanket	Sat. Time (s)	Soak Time (s)	Elute Time (s)
7	Elute Sample Container	Acetone	20	20	4	Off	2	180	180
8	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	180	180
9	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	120	120
10	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	120	120
11	Elute Sample Container	$MeCl_2$	17	15	6	Off	2	120	180

Step	Operation	Message	Attachment
12	Pause with Message	Part 2 of 3: Ion Exchange Elution. Remove the 250 mL collection flask containing the neutrals and acids elution. Stopper the flask and set aside for part 3. Then install a clean 125 mL flask to collect the ion exchange elution. Click “Continue” to Start Part 2.	None

			(mL)	Time (s)	Rate (#)	Blanket	Time (s)	Time (s)	Time (s)
13	Elute Sample Container	Acetone	20	20	4	Off	2	0	180
14	Elute Sample Container	1% NH_4OH	20	30	4	Off	2	120	120
15	Elute Sample Container	Acetone	20	20	4	Off	2	180	120
16	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	180	180
17	Elute Sample Container	$MeCl_2$	16	15	4	Off	2	120	180
18	Elute Sample Container	$MeCl_2$	16	15	4	Off	2	120	180
19	Elute Sample Container	$MeCl_2$	16	15	6	Off	2	120	180

Step	Operation	Message	Attachment
20	Pause with Message	Part 3 of 3: Carbon Cartridge Elution. Remove the carbon cartridge from the tubing lines. Connect the tubing ends together. Using a 20 cc syringe, plunge the carbon cartridge with air through the cap adapter to reseat the carbon bed on the frit. Replace the cap adapter with the funnel on the cartridge. Replace the disk holder with the cartridge. Replace the 125 mL flask with the 250 mL flask containing the neutrals and acids elution from Part 1. Stopper the 125 mL flask. Click “Continue” to start part 3.	None

Step	Operation	Dry Time (s)	Pump Rate	N2
21	Air Dry Disk Timer	60	6	Off

Step	Operation	Solvent	Solvent Vol. (mL)	Purge Time (s)	Pump Rate (#)	N2 Blanket	Sat. Time (s)	Soak Time (s)	Elute Time (s)
22	Elute Sample Container	Acetone	25	20	4	Off	3	60	60
23	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
24	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
25	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
26	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
27	Elute Sample Container	$MeCl_2$	17	15	6	Off	3	60	60

The samples were measured using GC/MS (6890GC/5975CMS, Agilent Technology). Operational conditions are shown in Table 3. All spiking standard solutions used were from Supelco, Bellefonte, PA. The surrogate mixes were from Restek Corp., Bellefonte, PA. Samples were spiked with 50 μ/L of analytes.

Results and Discussion

Prior to implementation of the sample preparation method, Section 9.4 requires each laboratory to complete an Initial Demonstration of Proficiency (IDP) by performing at least four replicate reference samples, taken through the entire Vacuum -7 in. Hg sample preparation and analysis steps. EPA Method 8270E key to analyte list in Section 1.1 suggests that method operation is considered appropriate if average recovery falls between 50–150 %. This note explains that actual recoveries may vary depending on the sample matrix, number of constituents being analyzed concurrently, analytical instrumentation, and the preparation method used. The note also includes a list of compounds that have historically been considered problematic (Section 1.4). Section 13.1 suggests that performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method, and use the method criteria is for guidance purposes only.

Table 4 presents data obtained for 4 consecutive runs completed as an IDP study per Section 9.4 with the use of Biotage suite of automated sample preparation equipment. For all compounds except for those of which EPA Method 8270E considers to be problematic (e.g., pyridine, NDMA, hexachlorocyclopentadiene and 2-picoline), average recovery fell in the 50–150% recovery range, and most were better, showing this preparation technique to be acceptable. Standard Deviation for this set of data was found to be 0.77–13 demonstrating reproducibility of results. Blank results are also shown (surrogate recoveries not shown) to indicate the low level of contamination in the process.

Matrix effects on method performance is documented by the Mode Consistent Flow analysis of an unspiked duplicate pair or a matrix spike (MS) and Oven Program matrix spike duplicate (MSD). Since the groundwater samples spike and matrix spike duplicate pair was performed as indicated in the method. Results for the MS/MSD are shown in Table 5 and MS Ions Monitored Scan masses 35–550 meet the criteria listed in Method 8270E for compliance having average percent recoveries fall within 50–150% for all compounds analyzed; except those of which EPA Method 8270E considers to be problematic. Relative Percent Difference (RPD) between the data sets was observed to be well below the generally accepted 20% RPD, except for one pair of hexachlorocyclopentadiene (light sensitive) MS/MSD pairs, where the RPD was 25%.

The data presented is normalized to demonstrate the extraction efficiency of compounds in EPA Method 8270E and to eliminate any external sources of error from the analytical instrumentation.

Table 2. DryVap® System Conditions.

Parameter	Setting
Dry Volume	200 mL
Heat Power	
Heat Timer	OFF
Auto Rinse Mode	OFF
Nitrogen Sparge	20 psi
Vacuum	-7 in. Hg

Table 3. Agilent 6890/5795MS.

Injection	
Volume	1 μL
Inlet Temperature	280°C
Mode	Splitless
Gas Type	Helium
Column Conditions	Zebron™ ZB-Semivolatiles (Phenomenex)
Mode	Consistent Flow
Oven Program	45°C hold for 1 min to 270°C at 15°C/min then to 318 °C at 6 °C/min
MS Ions Monitored	Scan masses 35-550

Table 4. Initial Demonstration of Proficiency (IDP).

Compounds:	% Rec	% Rec	% Rec	% Rec	Average % Recovery	SD
1,2,4,5-Tetrachlorobenzene	72.0	72.1	68.1	73.2	71.4	2.2
1,2,4-Trichlorobenzene	72.0	71.2	68.9	70.4	70.6	1.3
1,2-Dichlorobenzene	59.6	61.7	58.7	61.4	60.4	1.4
1,3,5,-Trinitrobenzene	87.0	85.6	86.9	89.7	87.3	1.7
1,3-Dichlorobenzene	53.9	57.8	55.2	57.1	56.0	1.8
1,3-Dinitrobenzene	91.2	88.6	91.9	93.2	91.2	1.9
1,4-Dichlorobenzene	56.4	60.0	56.6	59.5	58.1	1.9
1,4-Naphthoquinone	86.0	76.2	74.2	71.0	76.9	6.5
1-Naphthylamine	99.5	84.4	96.0	91.9	93.0	6.5
2,3,4,6-Tetrachlorophenol	93.2	90.8	91.3	94.3	92.4	1.6
2,4,5-Trichlorophenol	89.6	87.5	88.9	91.1	89.3	1.5
2,4,6-Tribromophenol	93.3	92.6	94.3	95.2	93.8	1.2
2,4,6-Trichlorophenol	91.0	88.9	89.5	91.8	90.3	1.3
2,4-Dichlorophenol	91.0	89.0	90.2	90.5	90.2	0.83
2,4-Dimethylphenol	87.5	83.6	87.7	87.7	86.6	2.0
2,4-Dinitrophenol	80.0	79.7	79.5	84.4	80.9	2.3
2,4-Dinitrotoluene	93.4	91.4	93.2	94.4	93.1	1.3
2,6-Dichlorophenol	89.5	87.2	87.7	90.0	88.6	1.4
2,6-Dinitrotoluene	95.4	91.4	95.0	94.9	94.2	1.9
2-Chloronaphthalene	82.9	81.4	81.4	82.5	82.1	0.77
2-Chlorophenol	79.9	78.4	77.3	78.4	78.5	1.1
2-Fluorobiphenyl	83.4	80.1	81.7	83.3	82.1	1.6
2-Fluorophenol	55.0	61.1	56.1	57.6	57.5	2.6
2-Methyl phenol	81.9	83.5	82.4	83.3	82.8	0.77
2-Methylnaphthalene	81.2	80.4	79.8	80.9	80.6	0.61
2-Naphthylamine	124	111	118	121	118	5.6
2-Nitroaniline	93.5	93.1	95.5	96.4	94.6	1.6
2-Nitrophenol	79.5	79.4	77.3	80.6	79.2	1.4
2-Picoline	44.6	48.5	45.0	47.9	46.5	2.0
3,3'-Dichlorobenzidine	107	97.3	102	106	103	4.2
3,3'-Dimethylbenzidine	129	109	114	126	119	9.6
3+4 Methyl phenol	83.7	85.4	85.1	86.9	85.3	1.3
3-Methylcholanthrene	88.4	82.5	89.1	91.1	87.8	3.7
3-Nitroaniline	110	105	107	110	108	2.2
4 Aminobiphenyl	129	128	119	129	126	5.0
4,6-Dinitro-2-methylphenol	89.6	86.9	88.6	90.3	88.9	1.4
4-Bromophenyl phenyl ether	88.6	89.6	90.3	91.7	90.0	1.3
4-Chloro-3-methylphenol	93.0	92.0	94.2	95.9	93.8	1.7
4-Chloroaniline	124	119	120	124	122	2.9
4-Chlorophenyl phenyl ether	86.7	87.1	88.5	88.6	87.7	0.96
4-Nitroaniline	90.7	89.9	89.2	90.0	90.0	0.64
4-Nitrophenol	89.1	87.8	92.3	92.4	90.4	2.3

Compounds:	% Rec	% Rec	% Rec	% Rec	Average % Recovery	SD
4-Nitroquinoline-1-oxide	93.0	91.7	91.6	96.0	93.1	2.0
5-nitro-o-toluidine	103	99.3	102	104	102	2.1
7,12-Dimethylbenz(a)-anthracene	87.8	82.1	87.4	90.2	86.9	3.4
Acenaphthene	86.2	84.4	86.5	86.0	85.8	0.94
Acenaphthylene	87.5	85.4	86.7	87.1	86.7	0.89
Acetophenone	80.2	78.4	76.0	77.8	78.1	1.7
Acetylaminofluorene	102	100	100	102	101	0.97
Aniline	89.8	85.1	81.2	84.4	85.1	3.5
Anthracene	89.8	89.2	90.6	93.0	90.6	1.7
Azobenzene	91.8	89.7	91.4	92.0	91.2	1.0
Benz(a)anthracene	93.6	92.2	92.2	94.4	93.1	1.1
Benzidine	109.5	76.9	89.3	90.9	91.7	13
Benzo(a)pyrene	90.1	88.7	88.8	92.1	89.9	1.6
Benzo(b)fluoranthene	91.2	89.5	89.5	93.8	91.0	2.0
Benzo(ghi)perylene	94.2	91.5	91.9	95.3	93.2	1.8
Benzo(k)fluoranthene	94.0	92.1	91.4	92.5	92.5	1.1
Benzoic acid	61.2	68.7	62.9	68.5	65.3	3.8
Benzyl alcohol	84.8	82.9	83.4	82.6	83.4	0.97
Bis(2-chlorethoxy)methane	88.5	84.3	84.8	84.9	85.6	1.9
Bis(2-chloroethyl)ether	72.2	71.6	68.7	71.0	70.9	1.5
Bis(2chloroisopropyl)ether	75.2	74.1	71.0	73.2	73.4	1.8
Bis(2-ethylhexyl) phthalate	103	103	103	106	104	1.3
Butyl benzyl phthalate	98.7	97.4	98.5	99.3	98.5	0.77
Carbazole	95.4	93.6	95.4	95.0	94.8	0.86
Chrysene	92.6	93.3	91.0	94.6	92.9	1.5
cis-Isosafrole	85.9	84.0	83.8	84.4	84.5	0.96
Dibenz(ah)anthracene	92.9	88.0	86.7	90.6	89.6	2.7
Dibenzofuran	87.8	86.7	87.7	87.9	87.5	0.54
Diethyl phthalate	96.8	94.3	97.5	97.2	96.4	1.4
Dimethyl phthalate	95.0	92.6	94.8	94.8	94.3	1.1
Dimethylaminoazobenzene	97.3	93.4	97.3	95.9	96.0	1.8
Di-n-butyl phthalate	101	99.0	102	102	101	1.4
Di-n-octyl phthalate	101	101	102	103	102	0.98
Dinoseb	91.1	91.8	93.1	94.1	92.5	1.4
Diphenylamine	94.2	90.6	94.6	93.9	93.3	1.8
Ethylmethane Sulfonate	81.5	75.2	75.9	75.9	77.1	2.9
Fluoranthene	93.3	92.8	93.4	94.9	93.6	0.93
Fluorene	89.2	87.3	88.1	89.3	88.5	0.96
Hexachlorobenzene	88.3	90.0	89.3	92.6	90.0	1.8
Hexachlorobutadiene	54.3	54.4	50.6	54.4	53.4	1.9
Hexachlorocyclopentadiene	49.2	45.9	39.2	40.6	43.7	4.6
Hexachloroethane	53.9	58.4	53.9	57.2	55.8	2.3
Hexachloropropene	54.7	55.1	52.3	54.7	54.2	1.3
Indeno(1,2,3-cd)pyrene	91.5	90.0	89.0	94.1	91.1	2.2

Compounds:	% Rec	% Rec	% Rec	% Rec	Average % Recovery	SD
Isophorone	86.2	83.2	82.6	83.6	83.9	1.6
Methapyrilene	89.5	87.0	89.0	88.8	88.6	1.1
Methyl Methane Sulfonate	54.5	49.9	58.2	60.5	55.8	4.6
Naphthalene	77.0	75.4	73.6	75.3	75.3	1.4
NDMA	38.0	39.6	47.7	50.0	43.8	5.9
Nitrobenzene	78.0	76.9	73.9	76.4	76.3	1.7
Nitrobenzene-d5	79.0	77.0	74.5	77.2	76.9	1.8
N-Nitroso-diethylamine	69.8	69.0	67.2	69.1	68.8	1.1
N-nitroso-di-n-butylamine	89.9	89.0	90.6	91.2	90.2	0.94
N-nitroso-di-n-propylamine	80.9	77.7	77.0	79.5	78.8	1.8
N-Nitrosomethyl ethylamine	61.0	62.2	62.2	65.3	62.7	1.8
N-Nitroso-morpholine	88.2	84.9	86.0	88.0	86.8	1.6
N-Nitroso-piperidine	84.6	81.9	81.4	83.2	82.8	1.4
N-Nitroso-pyrrolidine	83.4	79.8	81.8	84.5	82.4	2.0
o-toluidine	102	95.1	91.3	97.1	96.3	4.3
Pentachlorobenzene	79.1	79.6	78.8	82.1	79.9	1.5
Pentachloroethane	55.5	60.2	56.9	59.8	58.1	2.3
Pentachloronitrobenzene	92.6	91.3	89.9	93.2	91.8	1.5
Pentachlorophenol	88.2	88.8	88.2	92.8	89.5	2.2
Phenacetin	93.6	94.8	95.0	96.6	95.0	1.2
Phenanthrene	90.1	90.2	91.3	92.6	91.0	1.2
Phenol	56.1	57.5	58.4	57.0	57.2	0.95
Phenol-d5	56.8	57.3	58.1	57.7	57.5	0.56
p-Terphenyl-d14	93.9	91.9	89.3	93.9	92.3	2.2
Pyrene	93.1	91.6	92.7	94.2	92.9	1.1
Pyridine	31.4	36.1	32.5	35.6	33.9	2.3
Safrole	88.1	84.3	87.5	86.3	86.5	1.7
trans-Isosafrole	85.8	87.2	85.3	87.2	86.4	1.0

Table 5. Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Results - Ground Water.

	MS-1	MSD-1			MS-2	MSD-2		
Compounds:	Rec	% Rec	Avg (%)	RPD	% Rec	% Rec	Avg (%)	RPD
1,2,4,5-Tetrachlorobenzene	78.8	65.1	72.0	9.5	72.1	72.7	72.4	0.43
1,2,4-Trichlorobenzene	73.5	65.0	69.2	6.1	70.9	72.9	71.9	1.4
1,2-Dichlorobenzene	61.8	54.9	58.4	5.9	61.6	66.8	64.2	4.1
1,3,5,-Trinitrobenzene	91.1	85.4	88.2	3.2	84.0	84.0	84.0	0.04
1,3-Dichlorobenzene	56.8	50.2	53.5	6.2	57.4	62.7	60.0	4.4
1,3-Dinitrobenzene	96.6	88.7	92.7	4.3	89.1	86.1	87.6	1.7
1,4-Dichlorobenzene	59.0	51.4	55.2	6.9	59.1	63.9	61.5	3.9
1,4-Naphthoquinone	86.7	72.0	79.4	9.3	76.8	75.1	76.0	1.1
1-Naphthylamine	103	89.5	96.2	7.0	85.3	81.5	83.4	2.3
2,3,4,6-Tetrachlorophenol	94.1	87.0	90.6	3.9	89.0	88.3	88.7	0.41
2,4,5-Trichlorophenol	89.8	85.9	87.9	2.3	87.4	86.1	86.8	0.70
2,4,6-Tribromophenol	95.6	90.8	93.2	2.6	90.2	91.9	91.1	0.94
2,4,6-Trichlorophenol	91.9	86.2	89.1	3.2	88.6	86.9	87.8	0.98
2,4-Dichlorophenol	92.6	86.2	89.4	3.6	87.7	87.9	87.8	0.13
2,4-Dimethylphenol	90.0	83.9	86.9	3.5	86.6	84.8	85.7	1.1
2,4-Dinitrophenol	82.5	76.5	79.5	3.8	77.4	76.9	77.1	0.35
2,4-Dinitrotoluene	96.1	88.1	92.1	4.3	88.6	87.4	88.0	0.69
2,6-Dichlorophenol	90.6	85.1	87.8	3.1	87.8	87.6	87.7	0.14
2,6-Dinitrotoluene	95.5	89.8	92.7	3.1	90.5	88.6	89.6	1.1
2-Chloronaphthalene	85.9	76.7	81.3	5.6	80.4	80.6	80.5	0.12
2-Chlorophenol	80.8	75.0	77.9	3.7	78.4	82.2	80.3	2.4
2-Fluorobiphenyl	87.6	79.0	83.3	5.2	83.6	83.1	83.3	0.29
2-Fluorophenol	71.5	58.6	65.1	9.9	66.7	66.3	66.5	0.36
2-Methyl phenol	87.4	81.9	84.7	3.3	84.1	84.7	84.4	0.34
2-Methylnaphthalene	83.4	76.8	80.1	4.1	79.3	80.7	80.0	0.85
2-Naphthylamine	127	112	120	6.5	109	108	108	0.06
2-Nitroaniline	97.5	90.5	94.0	3.7	92.3	89.6	91.0	1.5
2-Nitrophenol	83.5	75.3	79.4	5.1	81.5	81.5	81.5	0.00
2-Picoline	47.0	42.0	44.5	5.6	47.6	49.6	48.6	2.0
3,3'-Dichlorobenzidine	109	102	105	3.3	99.9	98.2	99.1	0.88
3,3'-Dimethylbenzidine	129	117	123	5.0	110	102	106	3.7
3+4 Methyl phenol	89.8	83.7	86.8	3.5	84.5	86.3	85.4	1.0
3-Methylcholanthrene	90.4	84.7	87.5	3.3	80.0	82.6	81.3	1.6
3-Nitroaniline	111	99	105	5.6	104	102	103	0.99
4 Aminobiphenyl	127	123	125	1.6	123	116	120	2.8
4,6-Dinitro-2-methylphenol	91.6	86.5	89.1	2.9	83.0	83.7	83.3	0.42
4-Bromophenyl phenyl ether	91.5	84.9	88.2	3.7	86.1	86.4	86.3	0.13
4-Chloro-3-methylphenol	96.7	90.1	93.4	3.5	89.5	90.2	89.9	0.42
4-Chloroaniline	129	117	123	5.0	118	117	117	0.51
4-Chlorophenyl phenyl ether	89.4	82.2	85.8	4.2	84.3	83.9	84.1	0.25

	MS-1	MSD-1			MS-2	MSD-2		
Compounds:	Rec	% Rec	Avg (%)	RPD	% Rec	% Rec	Avg (%)	RPD
4-Nitroaniline	95.4	85.7	90.6	5.3	87.1	85.3	86.2	1.0
4-Nitrophenol	90.7	90.1	90.4	0.3	86.2	88.3	87.2	1.2
4-Nitroquinoline-1-oxide	97.9	89.2	93.6	4.6	86.7	87.6	87.2	0.49
5-nitro-o-toluidine	105	96.2	101	4.6	95.7	93.1	94.4	1.3
7,12-Dimethylbenz(a)-anthracene	88.8	83.9	86.4	2.8	79.4	84.3	81.8	3.0
Acenaphthene	89.0	81.3	85.2	4.5	82.5	83.1	82.8	0.33
Acenaphthylene	88.8	82.3	85.5	3.8	83.9	83.9	83.9	0.01
Acetophenone	80.7	72.8	76.7	5.1	77.4	80.9	79.2	2.2
Acetylaminofluorene	102	96.5	99.3	2.8	96.8	96.8	96.8	0.00
Aniline	91.9	79.2	85.5	7.4	84.7	86.0	85.3	0.77
Anthracene	92.9	84.7	88.8	4.6	86.2	87.1	86.6	0.52
Azobenzene	93.1	86.0	89.6	4.0	87.7	86.9	87.3	0.47
Benz(a)anthracene	92.8	88.4	90.6	2.5	89.0	90.3	89.6	0.71
Benzidine	103	88.1	95.5	7.7	70.4	80.2	75.3	6.5
Benzo(a)pyrene	90.3	85.4	87.9	2.8	85.6	86.9	86.3	0.77
Benzo(b)fluoranthene	92.3	86.4	89.4	3.3	87.4	88.7	88.0	0.74
Benzo(ghi)perylene	93.6	87.6	90.6	3.3	90.8	90.7	90.8	0.07
Benzo(k)fluoranthene	94.0	88.6	91.3	3.0	88.4	89.8	89.1	0.81
Benzoic acid	71.2	63.9	67.6	5.4	70.4	64.7	67.6	4.2
Benzyl alcohol	85.4	74.9	80.1	6.5	82.2	84.0	83.1	1.1
Bis(2-chlorethoxy)methane	88.6	82.1	85.3	3.8	83.7	85.8	84.8	1.2
Bis(2-chloroethyl)ether	72.0	64.4	68.2	5.6	70.1	76.4	73.3	4.3
Bis(2chloroisopropyl)ether	75.3	67.2	71.3	5.7	72.6	78.1	75.4	3.7
Bis(2-ethylhexyl) phthalate	101	98.0	99.5	1.5	95.7	98.2	97.0	1.3
Butyl benzyl phthalate	97.5	93.3	95.4	2.2	92.3	94.9	93.6	1.4
Carbazole	98.0	90.8	94.4	3.8	90.8	91.1	91.0	0.21
Chrysene	92.9	86.8	89.9	3.4	88.3	90.1	89.2	1.0
cis-Isosafrole	88.1	80.7	84.4	4.4	82.2	84.3	83.3	1.2
Dibenz(ah)anthracene	87.4	83.2	85.3	2.5	85.6	85.9	85.7	0.20
Dibenzofuran	90.2	83.3	86.7	3.9	84.1	84.5	84.3	0.19
Diethyl phthalate	98.9	92.2	95.6	3.5	91.4	91.2	91.3	0.088
Dimethyl phthalate	96.6	89.6	93.1	3.7	90.5	89.3	89.9	0.66
Dimethylaminoazobenzene	96.7	93.9	95.3	1.5	89.8	92.8	91.3	1.7
Di-n-butyl phthalate	102	97.5	99.7	2.2	95.6	96.1	95.9	0.26
Di-n-octyl phthalate	99.5	95.3	97.4	2.2	93.7	96.8	95.2	1.6
Dinoseb	94.4	91.7	93.1	1.5	85.8	89.4	87.6	2.1
Diphenylamine	95.8	88.9	92.3	3.7	89.4	88.7	89.1	0.38
Ethylmethane Sulfonate	78.7	72.0	75.3	4.5	75.0	81.6	78.3	4.2
Fluoranthene	95.9	88.1	92.0	4.2	89.8	90.1	89.9	0.17
Fluorene	89.4	83.5	86.5	3.4	85.0	84.5	84.8	0.27
Hexachlorobenzene	91.0	84.6	87.8	3.6	87.0	87.0	87.0	0.023
Hexachlorobutadiene	63.1	47.6	55.4	14	55.1	58.7	56.9	3.1

	MS-1	MSD-1			MS-2	MSD-2		
Compounds:	Rec	% Rec	Avg (%)	RPD	% Rec	% Rec	Avg (%)	RPD
Hexachlorocyclopentadiene	64.3	38.5	51.4	25	46.8	46.8	46.8	0.021
Hexachloroethane	56.0	49.2	52.6	6.5	55.9	61.9	58.9	5.1
Hexachloropropene	60.5	50.8	55.6	8.7	56.2	59.6	57.9	2.9
Indeno(1,2,3-cd)pyrene	91.6	86.4	89.0	2.9	88.4	89.4	88.9	0.54
Isophorone	86.5	79.7	83.1	4.1	82.8	83.3	83.0	0.31
Methapyrilene	90.0	84.9	87.5	2.9	84.2	84.7	84.5	0.28
Methyl Methane Sulfonate	55.6	53.2	54.4	2.2	48.9	64.2	56.6	14
Naphthalene	79.1	70.4	74.8	5.8	76.3	78.0	77.2	1.2
NDMA	41.1	39.6	40.4	1.9	37.4	52.4	44.9	17
Nitrobenzene	78.2	72.2	75.2	4.0	75.8	79.6	77.7	2.4
Nitrobenzene-d5	80.2	73.3	76.7	4.5	78.8	81.5	80.1	1.7
N-Nitroso-diethylamine	71.1	62.6	66.9	6.3	68.4	75.1	71.8	4.7
N-nitroso-di-n-butylamine	91.5	86.5	89.0	2.8	86.7	86.4	86.5	0.16
N-nitroso-di-n-propylamine	80.1	79.2	79.6	0.6	75.7	80.6	78.2	3.1
N-Nitrosomethyl ethylamine	62.1	54.9	58.5	6.2	61.7	70.1	65.9	6.3
N-Nitroso-morpholine	88.2	80.1	84.2	4.9	82.8	88.4	85.6	3.3
N-Nitroso-piperidine	84.4	78.3	81.3	3.8	79.6	82.8	81.2	2.0
N-Nitroso-pyrrolidine	83.7	79.9	81.8	2.3	78.7	82.3	80.5	2.3
o-toluidine	101	91.4	96.3	5.1	91.6	91.7	91.7	0.0
Pentachlorobenzene	83.3	73.7	78.5	6.1	77.2	78.8	78.0	1.0
Pentachloroethane	58.4	51.5	54.9	6.3	59.2	64.5	61.8	4.3
Pentachloronitrobenzene	94.1	87.2	90.7	3.8	87.5	87.7	87.6	0.13
Pentachlorophenol	90.7	86.0	88.4	2.7	85.9	86.6	86.3	0.39
Phenacetin	97.9	91.4	94.7	3.4	91.8	90.1	90.9	0.92
Phenanthrene	93.8	86.4	90.1	4.1	87.7	88.2	88.0	0.28
Phenol	68.7	56.2	62.5	10	59.8	62.0	60.9	1.8
Phenol-d5	69.3	57.0	63.2	9.8	62.2	63.4	62.8	1.0
p-Terphenyl-d14	92.1	86.9	89.5	2.9	89.5	90.4	89.9	0.47
Pyrene	94.8	86.7	90.7	4.4	89.2	89.3	89.2	0.045
Pyridine	32.2	29.9	31.1	3.7	34.6	35.2	34.9	0.86
Safrole	88.9	83.0	86.0	3.5	84.1	84.4	84.2	0.14
trans-Isosafrole	89.7	82.4	86.0	4.2	87.7	85.3	86.5	1.4

Conclusion

The precision and accuracy shown in the Initial Demonstration of Proficiency demonstrate that SPE combined with modern automation for extraction, extract drying and evaporation is an effective solution for a wide range of semivolatile compounds extracted at once from ground water samples. The results are compliant with the method requirements and show a solid base for laboratory use.

Solid phase extraction is an effective extraction technique that reduces the solvent that must be evaporated before GC/MS introduction, reducing costs and time for sample preparation. Disk formats, especially when used with the Fast Flow Disk holder can handle large amounts of particulate and will drastically reduce the possibility of emulsion formation, making the time for extraction more consistent.

This system can be extended to leachate preparation from soil samples or water with additional particulate content, such as wastewater. With today's modern analytical instrumentation the requirement for one liter samples may not be necessary for all projects and smaller sample (such as 100 mL) preparation has been demonstrated for semivolatile compounds.[3]

References

1. US EPA Method 8270E, https://www.epa.gov/sites/production/files/2017-04/documents/method_8260d_update_vi_final_03-13-2017_0.pdf .
2. US EPA Method 3535A, https://www.epa.gov/sites/production/files/2015-12/documents/3535a.pdf.
3. AN121-HOR, Solid Phase Extraction (SPE) in US EPA Method 625.1, the Performance of Smaller Samples, available at www.biotage.com.

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Literature Number: AN124-HOR.V.1

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