Application Note: 819

EPA Method 525.2: Extraction of Semivolatile Organic Compounds from Water Using AutoTrace 280 Solid-Phase Extraction Cartridges

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- Automated SPE
- EPA Method 525.2
- Semivolatiles
- Drinking Water
- AutoTrace 280
- GC
- GC/MS

Introduction

Traditionally, semivolatiles in drinking water have been extracted using liquid-liquid extraction techniques such as separatory funnel or continuous liquid-liquid extraction. While these techniques are effective, they use large volumes of organic solvent (up to 300 mL per sample) and require extensive user intervention or monitoring. Solid-phase extraction (SPE) is an alternative accepted extraction technique for USEPA Method 525.2 which uses significantly less solvent (up to 60 mL per sample). The Thermo Scientific Dionex AutoTrace 280 extraction instument offers an automated SPE technique that saves solvent and time, and decreases user intervention thanks to automation.

With the Dionex AutoTrace 280™ system, the compounds of interest are trapped on SPE adsorbents, then eluted with liquid solvents to generate an extract that is ready for analysis. The Dionex AutoTrace 280 system saves time, solvent, and labor ensuring high reproducibility and productivity for analytical laboratories. The unit can process up to six samples with minimal operator involvement. The Dionex AutoTrace 280 system uses powerful pumps (no check valves) and a proven positive-pressure technology to efficiently process liquid samples. Current analytical methods that may require SPE sample preparation include GC, GC-MS, LC, and LC-MS.

This work describes the use of the Dionex AutoTrace 280 instrument for extraction of semivolatile organic compounds from water samples.

Equipment

Dionex AutoTrace 280 Automated Large Volume SPE instrument for 6 mL Cartridges (Thermo Scientific PN 071385)

15 mL Conical Tubes (pack of 12) (Thermo Scientific PN 071056)

Required Solvents

Ethyl acetate (EtOAc)

Methylene chloride (CH_2Cl_2)

Methanol (MeOH)

Water

Standards

Restek (#31899) Semivolatiles Mix

Restek (#32415) OCP Mix AB #3

Restek (#33011) 525.2 Revised Chlorinated Pesticides Mix #2

Restek (#564292) Custom 525.2 Pesticide Std.

Restek (#565293) Custom 525.2 Misc Std.

Restek (#31825) Method 525.2 Internal Std Mix

Restek (#31826) Surrogate Mix

SPE Material

Thermo Scientific Dionex SolEx Cartridge: 6 mL, endcapped C18, 1 gram, Thermo Scientific (PN 074410)

Solvents

Solvent Bottle No.	Solvent Type
1	Water
2	MeOH
3	1:1 CH_2Cl_2:EtOAc
4	CH_2Cl^2
5	EtOAc

525.2 Solid-Phase Extraction Method

Enter the following method into the Dionex AutoTrace 280 program:

No.	Method	User Intervention
1	Process 6 samples using the following method steps	
2	Rinse Cartridge with 5.0 mL of EtOAc into Solvent Waste	
3	Rinse Cartridge with 5.0 mL of CH_2Cl_2 into Solvent Waste	
4	Condition Cartridge with 10.0 mL of MeOH into Solvent Waste	
5	Condition Cartridge with 10.0 mL of Water into Aqueous Waste	
6	Load 1100. 0mL of Sample onto Cartridge	
7	Pause and Alert operator, resume when CONTinue is pressed	– Check sample bottle to ensure all sample has been removed. – Add 5 mL EtOAc to each sample bottle and swirl to coat all sides of bottle. – Let stand.
8	Load 60.0 mL of Sample onto Cartridge	
9	Dry Cartridge with gas for 10.0 minutes	
10	Wash Syringe with 5.0 mL of MeOH	
11	Soak and Collect 4.0 mL Fraction using EtOAc	
12	Pause for 2.0 minutes	
13	Soak and Collect 4.0 mL Fraction using 1:1 CH_2Cl_2:EtOAc	
14	Pause for 2.0 minutes	
15	Collect 4.0 mL Fraction into collection vial using CH_2Cl_2	
16	Collect 4.0 mL Fraction into collection vial using CH_2Cl_2	
17	End	

Save and download this program on the Dionex AutoTrace 280 system for future use. Note: Up to 24 different programs can be saved onto the system. These methods can be written and transferred to the Dionex AutoTrace system via computer. Once the desired methods are stored on the Dionex AutoTrace 280 instrument, there is no further need for it to be connected to the computer.

Enter the following parameters for the Solid-Phase Extract under the Params Tab:

Flow Rates		SPE Parameters	
Cond Flow	40 mL/min	Push Delay	5 sec
Load Flow	20 mL/min	Air Factor	1.0
Rinse Flow	40 mL/min	Autowash Vol.	2.00 mL
Elute Flow	5 mL/min	**Instrument Parameters**	
Cond Air Push	15 mL/min	Max. Elution Vol.	16.0 mL
Rinse Air Push	20 mL/min	Exhaust Fan	Yes
Elute Air Push	5 mL/min	Beeper On	Yes

Clean Sample Path Method

It is advisable to clean the sample path after each batch of samples. The following is a suggested method that can be used:

No.	Method	User Intervention
1	Process 6 samples using the following method steps	
2	Clean Sample Path with 25 mL EtOAc into Solvent Waste	Place sample lines in EtOAc
3	Clean Sample Path with 25 mL MeOH into Solvent Waste	Place sample lines in MeOH
4	Clean Sample Path with 25 mL Water into Solvent Waste	Place sample lines in Water
5	Dry Cartridge with gas for 10.0 minutes	
6	End	

Results and Discussion

Pour the extracts through glass funnels containing a filter and 5–7 g of heat-treated sodium sulfate. Remove the sample bottles from the Dionex AutoTrace 280 instrument and pour the EtOAc that was used to rinse these bottles through the corresponding funnel containing sodium sulfate. Add 5 mL CH_2Cl to each sample bottle and swirl. Pour this through the corresponding funnel containing sodium sulfate. Concentrate the final sample extracts to 1 mL. Analyze by GC/MS.

The results listed in Table 1 show recoveries of semivolatile organic compounds from 4 spiked samples that were extracted using the AutoTrace 280 system.

Compound	Std. Conc.	Result	% Recovery	% RSD
Isophorone	1.000	0.977	97.7	2.21
1,3-Dimethyl-2-nitrobenzene	5.000	5.107	102.1	0.73
Naphthalene	1.000	0.995	99.5	3.36
Dichlorvos	1.000	1.058	105.8	4.81
Hexachlorocyclopentadiene	1.000	0.846	84.6	3.81
EPTC	1.000	1.023	102.3	3.25
Dimethylphthalate	1.000	1.000	100.0	2.95
Acenaphthylene	1.000	0.884	88.4	0.87
Choroneb	1.000	1.028	102.8	4.78
Acenaphthene	1.000	0.975	97.5	2.59
Molinate	1.000	1.037	103.7	1.96
Diethylphthalate	1.000	1.037	103.7	1.24
Propachlor	1.000	0.991	99.1	2.81
Fluorene	1.000	0.912	91.2	3.93
Trifluralin	1.000	0.878	87.8	1.27
alpha-BHC	1.000	0.984	98.4	1.85
Simazine	1.000	0.974	97.4	6.72
Hexachlorobenzene	1.000	0.852	85.2	2.96
Atrazine	1.000	1.055	105.5	2.68
beta-BHC	1.000	1.045	104.5	2.87
Pentachlorophenol	4.000	4.145	103.6	5.51
gamma-BHC	1.000	1.001	100.1	1.74
Terbacil	1.000	1.093	109.3	2.75
Chlorothalonil	1.000	1.065	106.5	2.25
Phenanthrene	1.000	0.967	96.7	2.61
delta-BHC	1.000	1.030	103.0	4.24
Anthracene	1.000	0.891	89.1	3.21
Acetochlor	1.000	1.043	104.3	1.65
Metribuzin	1.000	0.918	91.8	2.30
Alachlor	1.000	1.052	105.2	2.64
Prometryn	1.000	1.089	108.9	1.11
Di-n-butlyphthalate	1.000	1.088	108.8	1.03
Heptachlor	1.000	0.995	99.5	2.66
Bromacil	1.000	1.080	108.0	3.15
Malathion	1.000	1.237	123.7	3.36
Chlorpyrifos	1.000	1.044	104.4	1.61
Metolachlor	1.000	1.107	110.7	1.96
Thiobencarb	1.000	1.078	107.8	2.37
Ethyl-Parathion	1.000	1.028	102.8	4.63

Compound	Std. Conc.	Result	% Recovery	% RSD
DCPA (Dacthal)	1.000	1.031	103.1	3.67
Aldrin	1.000	0.914	91.4	1.55
Heptachlor Epoxide (isomer B)	1.000	1.044	104.4	2.29
Heptachlor Epoxide (isomer A)	1.000	1.084	108.4	3.41
Fluoranthene	1.000	1.013	101.3	3.79
Butachlor	1.000	1.126	112.6	2.35
alpha-Chlordane	1.000	0.980	98.0	1.20
gamma-Chlordane	1.000	0.983	98.3	1.61
Endosulfan I	1.000	1.113	111.3	4.07
Pyrene-d10	5.000	5.118	102.4	1.22
trans-Nonachlor	1.000	0.927	92.7	2.71
Pyrene	1.000	1.023	102.3	2.63
4,4′ DDE	1.000	0.910	91.0	3.80
Dieldrin	1.000	1.064	106.4	1.43
Chlorobenzilate	1.000	1.121	112.1	2.76
Endrin	1.000	1.050	105.0	9.31
4,4′ DDD	1.000	1.040	104.0	2.57
Endosulfan II	1.000	1.078	107.8	2.63
Endrin Aldehyde	1.000	1.149	114.9	2.64
Benzyl Butyl Phthalate	1.000	1.080	108.0	2.62
Di-(2-Ethylhexyl)adipate	1.000	0.852	85.2	1.20
4,4′ DDT	1.000	0.709	70.9	5.44
Endosulfan sulfate	1.000	1.091	109.1	4.66
Triphenlyphosphate	5.000	5.600	112.0	1.87
Methoxychlor	1.000	1.035	103.5	0.20
Endrin Keytone	1.000	1.269	126.9	2.28
Di (2-Ethylhexyl) Phthalate	1.000	0.963	96.3	1.15
Benz (a) anthracene	1.000	0.935	93.5	2.29
Chrysene	1.000	0.983	98.3	1.72
cis-Permethrin	1.000	0.808	80.8	0.69
Di-n-octylphthalate	1.000	0.936	93.6	1.79
trans-Permethrin	1.000	0.802	80.2	1.93
Benzo (b) fluoranthene	1.000	0.861	86.1	5.31
Benzo (k) fluoranthene	1.000	0.824	82.4	3.37
Benzo (a) Pyrene	1.000	0.779	77.9	2.82
Perylene-d12	5.000	4.614	92.3	3.04
Indeno (1,2,3-cd) pyrene	1.000	0.900	90.0	4.08
Dibenzo (a,h) anthracene	1.000	0.845	84.5	7.86
Benzo (g,h,i) perylene	1.000	0.839	83.9	5.58

Table 1. Recoveries of OCPs from water using the Dionex AutoTrace 280 system.

Conclusions

The Dionex AutoTrace 280 instrument with the cartridge configuration provides an automated, solid-phase extraction system that replaces traditional liquid-liquid extraction. Results show that using the cartridge configuration gives excellent recovery and precision results.

Acknowledgements

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List of Manufacturers

Thermo Fisher Scientific, 2000 Park Lane, Pittsburgh, PA 15275-1126 USA, Tel: 800-766-7000, www.fishersci.com.

Restek, 110 Benner Circle, Bellefonte, PA 16823 USA, Tel: 800-356-1688
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