Application Note

Analysis of Semi-Volatile Organic Compounds in Drinking Water Using EPA Method 525.3 with Semi-Automated Solid Phase Extraction (EZSpe®)

Introduction

EPA Method 525.3 outlines the procedure for the extraction and analysis of a wide range of organic compounds in water. The extraction method outlines the use of solid phase extraction for water matrix samples employing both cartridges and disks. Consistent with other EPA 500 series methods, EPA 525.3 incorporates a rigid set of QC and acceptance criteria requiring precise and reproducible analytical practices. The potential for error and the variability associated with manual extractions makes the benefits of semi-automating these processes apparent.

To meet demands for a low cost method that requires less financial investment than fully automated systems, FMS developed a simple semi - automated system which is fast, inexpensive and yields high quality data.
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Instrumentation

- FMS EZSpe™ System
- FMS SuperVap®
- Vacuum pump
- Thermo Trace GC w/DSQ MS and AS3000 Autosampler

Consumables

- FMS, Inc. 1 g SDVB cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Fisher 6 N Hydrochloric Acid
- Fisher Pesticide Grade Ethylacetate
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Dichloromethane
- Fisher Pesticide Grade Ethylacetate
- Restek 525.3 spiking standards

Procedure

- 6 samples (1L water each) are prepared and acidified with 2 mL HCl till pH ~ 2
- Spike with various 525.3 standards
- Put sample bottles in place and fill rinse bottles with 10 mL water
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 10 mL methanol (1 min soak) and 10 mL water (keep wet)
- Samples are loaded across cartridges under vacuum
- Rinse sample bottles with 10 mL water and load across cartridges
- Cartridges are dried under vacuum for 10 min
- Fill rinse bottles with 5 mL ethylacetate and rinse sample bottles automatically

Stage 2:

- Ethyl acetate from sample bottles is loaded across the cartridges (5 mL, 1 min soak) and the eluent is collected for analysis into Direct to GC Vial Collection Vessels
- Do same with 5 mL dichloromethane (first rinse sample bottles, then load across cartridges with 1 min soak)
- Extracts are dried over sodium sulfate or in line cartridges can be used downstream from SDVB cartridges

FMS SuperVap®

- Pre-heat temp: 40 ºC
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 40 ºC under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis on low resolution GC/MS

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Table 1 with average recoveries (%) for a number of 525.3 compounds at 25 ug/L

isophorone	102	Atrazine	103
1,3-Dimethyl-2-Nitrobenzene	100	Bromacil	103
hexachlorocyclopentadiene	85	di-n-butylphthalate	108
Vernolate	102	Metolachlor	103
Butylate	104	Aldrin	83
Pebulate	106	DCPA	99
dimethylphthalate	73	2,2',4,4'-tetrachlorobipheny	88
Etridiazole	73	Diphenamid	104
Chloroneb	101	MGK_(A)	107
2,6-dinitrotoluene	84	Heptachlor_epoxide	99
molinate	75	Gamma-Chlordane	84
diethylphthalate	77	Butachlor	102
Fluorene	92	Alpha_Chlordane	85
2,4-dinitrotoluene	98	Pyrene	81
Propachlor	86	Endosulfan_I	96
Cycloate	98	Trans-nanochlor	88
Mevinphos	93	Napropamide	105
Chlorpropham	84	Endrin	129
Gamma-BHC	93	Dieldrin	87
Beta-BHC	91	2,2',3',4,6-pentachlorobiphenyl	87
Alpha-BHC	93	2,2',4,4',5,6'-hexachlorobiphenyl	72
2,3-dichlorobiphenyl	94	Chlorobenzilate	89
Ethoprop	89	Endosulfan_II	90
Hexachlorobenzene	83	DDD	73
Atraton	101	Norflurazon	76
Prometon	101	Butyl_benzyl_phthalate	89
Simizine	93	Endosulfan_Sulfate	78
Propazine	77	DDT	74
terbufos	106	Hexazinone	87
Methyl_Paraoxon	92	bis(2-ethylhexyl)adipate	78
Pronamide	103	Triphenylphosphate	103
2,4,5-trichlorobipenyl	97	Benzo[a]anthracene	78
Phenanthrene	94	Methoxychlor	83
Disulfoton	121	2,2',3,3',4,4',6-heptachlorobiphenyl	83
Anthracene	91	bis(2-ethylhexyl)_phthalate	78
Alachlor	114	Fenarimol	93
Terbacil	116	Cis-Permethrin	78
Delta-BHC	100	Trans-Permethrin	76
Simetryn	91	Benzo[b]fluoranthene	81
Ametryn	91	Fluridone	108
Heptachlor	87	Indeno[1,2,3-cd]pyrene	88
Prometryn	82	Dibenzo[a,h]anthracene	86
Terbutryn	93	Benzo[g,h,i]perylene	85
Cyanazine	99		

Conclusions

Reviewing the sample data shows high recoveries for over 85 analytes, demonstrating excellent efficiency for all classes of compounds (acceptance window 70-130%). Samples can be taken from collection bottle to GC vial in one quick, consistent, reproducible process that will save laboratories both time and money.

FMS EZSpe system

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