Application Note

Automated Solid Phase Extraction of EPA 525.2, Semi-Volatile Organic Compounds in Drinking Water.

Introduction

EPA Method 525.2 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 525.2 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 automating these processes apparent.

The following procedure was developed to demonstrate the benefits of automating EPA Method 525.2 using Waters Oasis® HLB cartridges, the FMS SPE and the FMS SuperVap™ Direct-to-Vial Concentration system. This one step method delivers rapid, consistent and reproducible extraction and concentration of water samples for analysis by GC/MS.

Instrumentation and Consumables

- FMS, Inc. SPE System
- FMS, Inc. SuperVap™ Concentrator
- FMS, direct to vial concentrator tubes
- Thermo Certified GC/MS auto sampler vials
- 1 gram Waters Oasis® HLB Cartridge
- Restek 2 gram Sodium sulfate Cartridge
- Thermo Trace GC w/DSQ MS and AS3000 Autosampler

Reagents

- Fisher Optima* Methanol
- Fisher Optima* Methylene Chloride
- Fisher Optima* Ethyl Acetate
- Fisher HPLC Water
- Fisher Concentrated Sulfuric Acid
- Ultra Scientific Calibration/Spiking solutions (SVM-525, PPM-525E, NPM-525C, NPM-525B, NPM-108B, ISM-510)

Procedure:

Sample Prep

Five, one liter samples are measured out in glass sample collection bottles

Each sample is spiked with 1 mL of 525 spiking solution (dilute of Ultra Scientific standards, 5 ug/mL majority).

Sample PH adjusted to <2 with 1:1 sulfuric acid solution

10 mL Methanol added to each sample bottle

FMS SPE system

1. The HLB cartridges are conditioned with 10 mL Methanol
2. The cartridges are conditioned with 10 mL DI H_2O
3. The samples are loaded across the cartridges via vacuum (~75 mL/min)
4. Sample bottles are automatically rinsed with DI water and the rinse is loaded onto cartridge
5. The cartridges are dried with Nitrogen for 2 minutes
6. The cartridges are eluted with 10 mL of ethyl acetate
7. The cartridges are eluted with 10 mL methylene chloride
8. The cartridges are purged with a Nitrogen push

SuperVap Concentrator system

1. Pre-heat temp: 45 ºC
2. Pre-heat time: 20 minutes
3. Heat in Sensor mode: 45 ºC
4. Nitrogen Pressure: 10 PSI
5. End point: 1mL

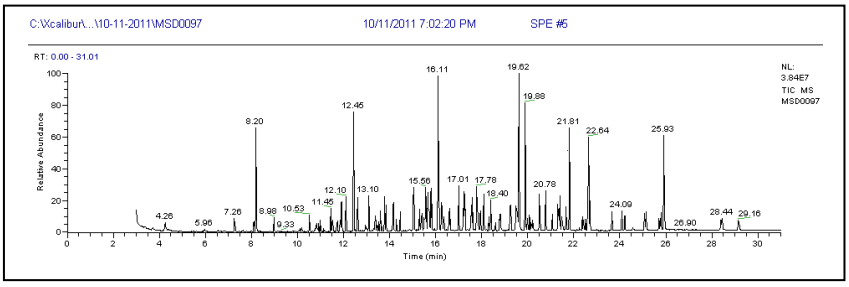
FMS
Fluid Management Systems

Figure 1: PowerPrep™ SPE and PowerVap™ Concentrator systems.

Compound	Spike Conc	Mean Rec	STD DEV
Terbufos	5 µg/L	107.8%	4.6%
Chrysene-d12	25 µg/L	91.1%	16.8%
Anthracene	5 µg/L	95.1%	2.3%
2,2',4,4',5,6'-hexachlorobiphenyl	5 µg/L	99.7%	13.4%
2-chlorobiphenyl	5 µg/L	101.9%	2.6%
Ethoprop	5 µg/L	120.2%	5.3%
Tricyclazole	5 µg/L	138.7%	9.1%
DDD	5 µg/L	109.0%	10.1%
Cis-Permethrin	5 µg/L	98.6%	9.1%
Benzo[a]pyrene	5 µg/L	100.8%	11.3%
Propochlor	5 µg/L	99.3%	4.5%
Pronamide	5 µg/L	118.4%	4.3%
Terbutryn	5 µg/L	89.6%	3.1%
Triademefon	5 µg/L	101.6%	3.7%
DEF	50 µg/L	70.4%	12.1%
DCPA	5 µg/L	114.7%	6.2%
DDE	5 µg/L	101.1%	10.6%
Acenaphthene-d10	25 µg/L	90.1%	11.5%
Pyrene	5 µg/L	105.7%	11.9%
2,4-dinitrotoluene	5 µg/L	110.2%	7.3%
Butylate	5 µg/L	119.5%	5.0%
Dieldrin	5 µg/L	101.1%	11.1%
Perylene-d12	25 µg/L	105.9%	12.4%
Dibenzo[a,h]anthracene	5 µg/L	117.9%	9.8%
Fenamiphos	5 µg/L	109.4%	7.1%
Alachlor	5 µg/L	106.8%	8.6%
Norflurazon	5 µg/L	127.7%	14.5%
Simizine	5 µg/L	123.0%	5.0%
Phenanthrene-d10	25 µg/L	96.8%	11.2%
Benzo[b]fluoranthene	5 µg/L	109.7%	12.5%
Chrysene	5 µg/L	110.3%	3.1%
2,2',3,3',4,5',6,6'-octachlorobiphenyl	5 µg/L	99.2%	13.5%
Chlorpyrifos	5 µg/L	101.6%	7.7%
Endrin_Aldehyde	5 µg/L	112.4%	22.5%
Phenanthrene	5 µg/L	104.8%	3.3%
bis(2-ethylhexyl)adipate	5 µg/L	106.1%	8.1%
Diethylphthalate	5 µg/L	112.4%	5.3%
hexachlorocyclopentadiene	5 µg/L	73.7%	7.2%
Diazinon	5 ug/L	97.4%	3.5%
Pebulate	5 ug/L	106.4%	6.0%
Cyanazine	5 ug/L	100.7%	6.2%
2,2',3,3',4,4',6-heptachlorobiphenyl	5 ug/L	103.4%	15.2%
Indeno[1,2,3-cd]pyrene	5 ug/L	115.4%	9.3%
bis(2-ethylhexyl)phthalate	5 ug/L	109.1%	15.5%
Isophorone	5 ug/L	108.4%	4.0%
Disulfoton	5 ug/L	91.0%	5.5%
Mevinphos	5 ug/L	109.3%	6.8%
Pyrene-d10	25 ug/L	101.0%	14.4%
Tebuthiuron	5 ug/L	126.1%	8.6%
Trifluralin	5 ug/L	107.7%	3.4%
Ametryn	5 ug/L	122.7%	5.2%
Bromacil	5 ug/L	115.7%	1.9%
Fenarimol	5 ug/L	124.4%	15.9%
Heptachlor Epoxide	5 ug/L	111.9%	5.6%
Trans-nanochlor	5 ug/L	97.3%	7.3%
Benzo[a]anthracene	5 ug/L	99.7%	11.1%
Atrazine	5 ug/L	120.1%	7.7%
Trerbacil	5 ug/L	113.0%	13.4%
Etridiazole	5 ug/L	108.5%	4.2%
Alpha-BHC	5 ug/L	95.9%	2.4%
Beta-BHC	5 ug/L	105.5%	5.2%
Delta-BHC	5 ug/L	102.2%	5.2%
Alpha Chlordane	5 ug/L	100.6%	10.3%
Endrin	5 ug/L	120.2%	17.9%
Methoxychlor	5 ug/L	99.8%	15.8%
Acenaphthylene	5 ug/L	92.6%	13.0%
Pentachlorophenol	5 ug/L	108.2%	4.3%
2,6-dinitrotoluene	5 ug/L	111.5%	7.4%
Hexachlorobenene	5 ug/L	90.9%	2.8%
Atraton	5 ug/L	119.2%	2.5%
Propazine	5 ug/L	123.2%	3.6%
Napropamide	5 ug/L	110.4%	10.1%
Endosulfan I	5 ug/L	121.4%	4.1%
Benzo[k]fluoranthene	5 ug/L	114.9%	12.5%
Butylbenzylphthalate	5 ug/L	125.3%	14.0%
2,4,5-trichlorobiphenyl	5 ug/L	104.0%	2.6%
Dimethylphthalate	5 ug/L	105.5%	6.6%
EPTC	5 ug/L	109.4%	5.9%

Chloropham	5 µg/L	120.6%	6.7%
Metolachlor	5 µg/L	112.0%	4.3%
Stirofos	5 µg/L	111.9%	7.0%
Aldrin	5 µg/L	102.3%	7.0%
Trans-Permethrin	5 µg/L	107.8%	11.5%
2,2',3',4,6-pentachlorobiphenyl	5 µg/L	107.1%	9.3%
2,2',4,4'-tetrachlorobiphenyl	5 µg/L	97.5%	5.1%
Molinate	5 µg/L	110.0%	6.1%
Cycloate	5 µg/L	114.4%	3.3%
Simetryn	5 µg/L	123.8%	4.7%
MGK (B)	5 µg/L	120.5%	5.0%
Butachlor	5 µg/L	124.9%	3.6%
Chlorothalonil	5 µg/L	109.1%	4.3%
MGK (A)	5 µg/L	120.3%	0.6%
Gamma-Chlordane	5 µg/L	104.8%	8.2%
Triphenylphosphate	25 µg/L	119.0%	10.1%
Fluorene	5 µg/L	108.9%	6.3%
2,3-dichlorobiphenyl	5 µg/L	98.1%	1.1%
Carboxin	5 µg/L	75.0%	9.0%
Methyl Paraoxon	5 µg/L	118.6%	3.4%

Prometryn	5 µg/L	122.8%	6.1%
Fluridone	5 µg/L	144.4%	11.5%
Endosulfan Sulfate	5 µg/L	116.7%	16.1%
1-bromo-2-nitrobenzene	5 µg/L	91.4%	6.1%
1,3-dimethyl-2-nitrobenzene	25 µg/L	102.8%	2.7%
Benzo[g,h,i]perylene	5 µg/L	117.2%	9.5%
Di-n-butylphthalate	5 µg/L	116.2%	8.6%
Vernolate	5 µg/L	114.0%	6.1%
Diphenamid	5 µg/L	106.5%	6.0%
Dichlorovos	5 µg/L	116.8%	6.6%
Heptachlor	5 µg/L	97.9%	6.0%
Chlorobenzilate	5 µg/L	124.6%	9.1%
Choroneb	5 µg/L	114.9%	5.0%
Prometon	5 µg/L	119.1%	3.6%
Hexazinone	5 µg/L	122.4%	13.1%
Gamma-BHC	5 µg/L	116.6%	5.1%
Endosulfan II	5 µg/L	119.0%	15.9%
DDT	5 µg/L	93.4%	11.2%



Chromatogram of an extracted 525 sample on the DSQ

Conclusions

Reviewing the sample data shows high recoveries for over 100 spiked analytes, demonstrating excellent efficiency for all classes of compounds. Low deviations between runs shows good run-to-run reproducibility. The FMS SPE and SuperVap™ system are suitable for the full automation from sample to vial of EPA Method 525.2. Samples can be taken from collection bottle to GC vial in one quick, consistent, reproducible process that will save laboratories both time and money.

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