Application Note

Analysis of Selected Semivolatile Organics in Drinking Water with Semi-Automated Solid Phase Extraction (EZSpe®) Using EPA Method 526

Introduction

This application note describes the analysis of a number of semi volatile organics in drinking water samples using US EPA method 526. The method uses Solid Phase Extraction and is of particular interest to environmental laboratories.

To meet demands for a low cost method that requires less financial investment than 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 DSQ low resolution GC/MS

Consumables

- FMS, Inc. 1 g SDVB cartridge
- Ultra pure DI water
- L-Ascorbic Acid
- Trisodium-EDTA
- Diazolidinyl Urea
- Tris(hydroxymethyl)aminomethane
- Tris(hydroxymethyl)aminomethane hydrochloride
- Fisher Pesticide Grade Dichloromethane
- Fisher Pesticide Grade Ethyl Acetate
- Fisher Pesticide Grade Methanol
- 526 Standard Analyte Solutions

Procedure

- 6 samples (1L water each) add L-Ascorbic Acid, Trisodium-EDTA, Diazolidinyl Urea, Tris(hydroxymethyl)-aminomethane and Tris(hydroxymethyl)-aminomethane hydrochloride (pH ~ 7,buffered)
- Spike relevant standards
- Cartridges are installed in each of the six positions
- Fill rinse bottles with 5 mL EtAc

Stage 1:

- Vacuum is turned on
- Clean cartridges with 5 mL EtAc (1 min soak) and 5 mL DCM
- Cartridges are conditioned with 5 mL methanol (30 sec soak) and another 5 mL methanol (keep wet)
- Cartridges are conditioned with 2 x 5 mL water (keep wet)
- Samples are loaded across cartridges under vacuum at 20 mL/min
- Cartridges are dried under vacuum for 10 min
- Sample bottles are automatically rinsed from the rinse bottles with 5 mL EtAc

Stage 2:

- Elute cartridges with 5 mL EtAc from sample bottles (30 sec soak) and collect
- Same step for 5 mL dichloromethane, colect
- Extracts are dried over sodium sulfate or in line cartridges can be used downstream from SDVB cartridges. After extracts have been collected run 2 x 3 mL 50/50 DCM/EtAc across sodium sulfate and collect

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

Analysis

- Analysis done with low res GC/MS

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Table 1 with recoveries for 526 Analytes at 5 ug/L

Compound name	Average (%)
Nitrobenzene	74
2,4-Dichlorophenol	80
2,4,6-Trichlorophenol	88
1,2-Diphenylhydrazine	93
Prometon	96
Terbufos	80
Fonofos	88
Diazinon	87
Disulfoton	82
Acetochlor	85
Cyanazine	86

Conclusions

The results of these water samples demonstrate the ability of the FMS EZSpe system to deliver accurate and reliable results. Recoveries are well within the method's 70-130% acceptance window. The semi-automated EZSpe is superior to traditional, time-consuming, inconsistent and expensive liquid/liquid extractions.

References

US EPA Method 526

FMS EZSpe system

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