Application Note

Analysis of Explosives in Drinking Water Using EPA Method 529 with Semi-Automated Solid Phase Extraction (EZSpe®)

Introduction

EPA method 529 details the procedure for the extraction and analysis of explosives in finished drinking water. The method uses solid phase extraction of water samples to partition analytes of interest from aqueous samples, and analyzes them by GC/MS analysis.

The method calls for the extraction of 1 liter samples by Solid Phase Extraction (SPE) using a DVB cartridge or disk. Extracts are eluted with Ethyl Acetate and run on a GC/MS system. The use of a PTV injection system is required to optimize the loading of thermally labile compounds such as RDX.

The following details the use of the Fluid Management Systems, Inc EZ-Spe® semi-automated extraction system to execute the procedure according to EPA 529.

Instrumentation

- FMS EZSpe® System
- FMS SuperVap®
- Vacuum pump
- Waters Acquity UPLC
- Waters Xevo TQD

Consumables

- FMS, Inc. 0.5 g charcoal cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Ammonium acetate
- Fisher Pesticide Grade Methanol
- Restek 535 spiking standards

Procedure

- 6 samples (250 mL water each) are prepared
- Spike with various 535 standards
- Put sample bottles in place and fill rinse bottles with 15 mL 10 mM ammonium acetate in methanol
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 2 x 10 mL ammonium acetate/methanol (keep wet) and 3 x 10 mL water (keep wet)
- Samples are loaded across cartridges under vacuum at 10 mL/min
- Cartridges are rinsed with 5 mL water and dried with nitrogen for 3 minutes.
- Sample bottles are automatically rinsed from the rinse bottles with 15 mL ammonium acetate/methanol.

Stage 2:

- Ammonium acetate/methanol from sample bottles is loaded across the cartridges (5 mL, then 10 mL) and the eluent is collected for analysis into Direct to LC Vial Collection Vessels

FMS SuperVap®

- Pre-heat temp: 65 ºC
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 65 ºC under nitrogen (7-10 psi)
- Direct to LC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

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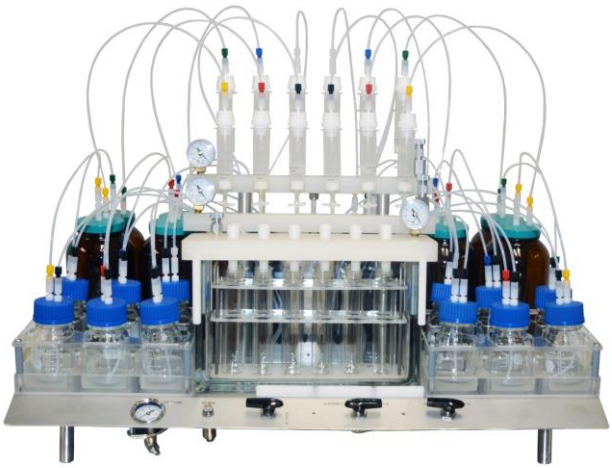
Table 1 with recoveries for a number of 529 compounds

Compound	%Recovery	Stdev
Nitrobenzene	97.1	3.0
2-Nitrotoluene	98.8	3.8
3-Nitrotoluene	99.3	3.0
4-Nitrotoluene	101.3	5.5
1,3-Dinitrobenzene	106.8	1.5
2,6-Dinitrotoluene	95.6	2.5
2,4-Dinitrotoluene	90.5	2.8
1,3,5-Trinitrobenzene	80.9	2.0
2,4,6-Trinitrotoluene	77.4	2.5
RDX	104.6	4.7
4-Amino-4,6-dinitrotoluene	91.9	2.0
3,5-Dinitroaniline	88.4	1.5
2-Amino-4,6-dinitrotoluene	94.8	2.4
Tetryl	77.5	5.7

Conclusions

The final analysis of the sample replicates yielded recoveries well within the 70-130% limits defined in the method for all analytes. Variation between samples replicates resulted in single digit deviations demonstrating excellent reproducibility. Semi-automated Sample Preparation produce consistent, accurate results and make the FMS TurboTrace SPE® and SuperVap® Concentration system an ideal solution for drinking water labs currently performing EPA 529 extractions manually.



FMS EZSpe® System

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