

EPA 529; Explosives in Drinking Water by Solid Phase Extraction (SPE)



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 TurboTrace™ SPE extraction system to fully automate the extraction procedure contained in EPA 529.

Instrumentation and Consumables

Instrumentation

- FMS, Inc. TurboTrace™ Solid Phase Extraction System
- FMS, Inc. SuperVap™ 12 Position Concentrator
- FMS, direct-to-vial concentrator tubes
- Thermo Trace Ultra GC with PTV Injection Port
- Thermo Polaris Q Mass Spectrometer

Consumables

- FMS DVB Cartridge (1 gram)
- Fisher Optima Grade Methanol
- Fisher Optima Grade Ethyl Acetate
- Fisher HPLC Grade Water
- Fisher Anhydrous Sodium Sulfate

Procedure

TurboTrace SPE System Program

1. Cartridges conditioned with 10 mL MeOH
2. Cartridges conditioned with 10 mL H₂O
3. Samples loaded across cartridges via vacuum
4. Cartridges dried under vacuum for 5 minutes.
5. Sample bottle sprayed with 20 mL Ethyl Acetate.
6. 20 mL Ethyl Acetate spray loaded across cartridge and collected.
7. Cartridges eluted with 5 mL Ethyl Acetate
8. Eluted solvent nitrogen purged directly into FMS SuperVap for concentration**.
9. Total Time ~30 minutes

**Removal of residual water from extract by in-line sodium sulfate filtration.

SuperVap Concentrator

1. Preheat temp: 20 minutes at 45 °C
2. Evap mode w/Sensor temp: 45 °C
3. Nitrogen Pressure: 10 PSI



Figure 1; FMS, Inc TurboTrace SPE system with the SuperVap concentrator.



Results

Table 1; Results for 1 liter replicates spiked at 1 µg/L.

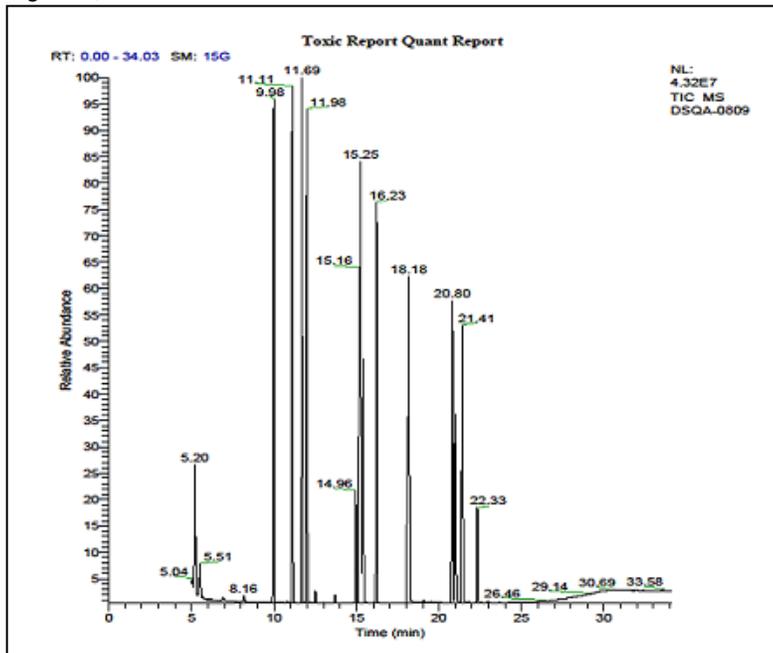
Compound	Mean Rec.	STD DEV
Nitrobenzene	97.1%	3.0%
2-Nitrotoluene	98.8%	3.8%
3-Nitrotoluene	99.3%	3.0%
4-Nitrotoluene	101.3%	5.5%
1,3-Dinitrobenzene	106.8%	1.5%
2,6-Dinitrotoluene	95.6%	2.5%
2,4-Dinitrotoluene	90.5%	2.8%
1,3,5-Trinitrobenzene	80.9%	2.0%
2,4,6-Trinitrotoluene	77.4%	2.5%
RDX	104.6%	4.7%
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.

Totally 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..

Figure 2; TIC for EPA 529 LCS



For more information contact FMS:
 FMS Inc.
 580 Pleasant Street
 Watertown, MA 02472
 Phone: (617) 393-2396
 Fax: (617) 393-0194
 Email: onlineinfo@fms-inc.com
 Web site: fms-inc.com