Application Note

Analysis of Halogenated Acetic Acids and Dalapon in Drinking Water Using EPA Method 552.1 with Automated Solid Phase Extraction (TurboTrace®)

Introduction

Haloacetic acids are known by-products of water chlorination. They often accumulate in significant quantities and have been implicated in multiple undesirable health outcomes by various epidemiological analyses.

EPA Method 552.1 outlines the procedure for the extraction and analysis of these compounds in drinking 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 552.1 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. Hence, FMS developed a simple automated system which is fast, inexpensive and yields high quality data.

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Instrumentation

- FMS TurboTrace® System
- FMS SuperVap®
- Vacuum pump
- Agilent 7890A GC FID/ECD

Consumables

- FMS, Inc. 1 g SDVB cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Fisher 6 N Hydrochloric Acid/Sodium Hydroxide
- Fisher 6 N sulfuric acid
- Fisher Pesticide Grade Methanol
- Fisher methyl tert-butyl ether
- Restek 552.1 spiking standards

Procedure

- 6 samples (100 mL water each) are prepared and acidified with HCl till pH ~ 5
- Spike with various 552.1 standards
- Put sample bottles in place and fill rinse bottles with 4 mL 10% sulfuric acid in methanol
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 10 mL methanol, 10 mL water, 10 mL 1M HCl in methanol, 10 mL water, 10 mL 1M NaOH, 10 mL water (keep wet after each addition)
- Samples are loaded across cartridges under vacuum, at 2 mL/min.
- Cartridges are dried under vacuum with 10 mL methanol
- Sample bottles are automatically rinsed from the rinse bottles with 4 mL 10% sulfuric acid in methanol

Stage 2:

- Sulfuric acid/methanol from sample bottles is loaded across the cartridges and
- To the eluent is added 2.5 mL of methyl tert-butyl ether, which is then heated for 1 hour and collected for analysis into Direct to GC Vial Collection Vessels
- Extracts are dried over sodium sulfate or in line cartridges can be used downstream from SDVB cartridges

FMS SuperVap®

- Pre-heat temp: 45 ºC
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 45 ºC under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

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Table 1 with recoveries for the 552.1 compounds

Compound	%Recovery	Stdev
Monochloroacetic acid	83.2	1.63
Dichloroacetic acid	91.4	2.75
Trichloroacetic acid	76.8	1.99
Monobromoacetic acid	88.2	1.92
Bromochloroacetic acid	96.6	1.84
Dibromoacetic acid	100.3	2.03
Dalapon	79.2	3.60

Conclusions

Reviewing the sample data shows high recoveries for the seven 552.1 analytes, demonstrating excellent efficiency for these compounds. 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 TurboTrace® System

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