

Analysis of Endothall in Drinking Water Using EPA Method 548.1 with Semi-Automated Solid Phase Extraction (EZSpe®)



Introduction

Endothall is a white, colorless organic solid used primarily as a defoliant and an herbicide. It is widely used to control invasive aquatic exotics like Eurasian milfoil. Regulated by the EPA under the Safe Water Drinking Act, it is classified as a Class II toxicant that can cause liver and kidney damage with prolonged exposure, and it can also affect organs of the digestive system.

This application note follows EPA method 548.1 using ion exchange Solid Phase Extraction cartridges to extract endothall from a water matrix. Traditionally, a lengthy manual process with many conditioning steps is required to use ion exchange cartridges. The following demonstrates how the FMS EZ-Spe system can automate the conditioning, extraction, and elution of samples in a single turnkey process.

Instrumentation & Consumables

- FMS EZSpe® System
- FMS SuperVap®
- Vacuum pump
- Thermo Trace GC Ultra
- Thermo Finnigan Trace DSQ
- 500 mg Waters Oasis Max cartridges
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Methylene Chloride
- Fisher Sulfuric Acid
- Fisher Sodium Hydroxide Pellets
- Fisher Anhydrous Sodium Sulfate
- Restek EPA 548.1 spiking standards

Procedure

- 6 samples (100 mL water each) are prepared
- Spike with various 548.1 standards
- Put sample bottles in place and fill rinse bottles with 8 mL of 10% sulfuric acid in methanol, then with 6 mL of dichloromethane
- 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 10% sulfuric acid in methanol, 20 mL sodium hydroxide, and 20 mL water.
- Samples are loaded across cartridges under vacuum, at 10 mL/min.
- Cartridges are dried under vacuum for 5 min (no nitrogen)
- Sample bottles are automatically rinsed from the rinse bottles with 8 mL of 10% sulfuric acid in methanol, then with 6 mL of dichloromethane

Stage 2:

- Sulfuric acid/methanol from sample bottles is loaded across the cartridges (8 mL)
- The above step is repeated for 6 mL of dichloromethane and the eluent is 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 C-18 cartridges

FMS SuperVap®

- Pre-heat temp: 40 °C
- Pre-heat time: 20 minutes
- Heat in Sensor mode at 40 °C under nitrogen (15 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis



Table 1 with recoveries and standard deviations of samples extracted and analyzed.

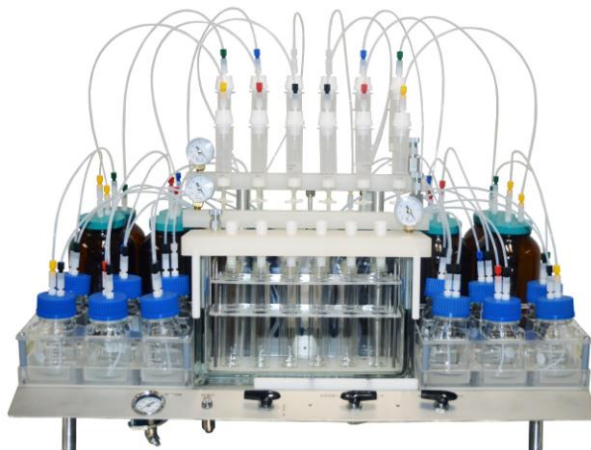
	Endothall Concentration	%Recovery
Sample #1	47.6 ug/mL	95.2
Sample # 2	49.3 ug/mL	98.6
Sample # 3	48 ug/mL	96.0
	Std Deviation	2.7

Derivatization/Partition

Following extraction, sample derivatization was conducted following section 11.4 of EPA method 548.1. Extracts were spiked with internal standard following the partition step and then returned to FMS SuperVap Concentrator equipped with direct-to-GC vial tubes. Following the above parameters, extracts were taken to a 1 mL final volume and transferred to GC/MS for analysis.

Conclusions

Reviewing the sample data shows high recoveries for endothall, demonstrating excellent efficiency. 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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FMS EZSpe® System