

Nota applicativa

Endothall in Drinking Water and Soil

Waters Corporation



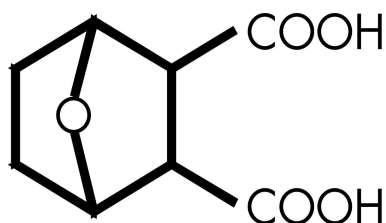
This is an Application Brief and does not contain a detailed Experimental section.

Abstract

This application brief highlights about analysis of endothall in drinking water and soil.

Introduction

The compound analyzed in this study is endothall.



ENDOTHALL

Experimental

LC-MS Method

| | |
|-------------------|--|
| Column: | Symmetry Shield RP8, 2.1 x 100 mm, 3.5 μ m |
| Part number: | WAT058969 |
| Mobile phase: | 5% Acetonitrile in 1% formic acid/water |
| Flow rate: | 200 μ L/min |
| Injection volume: | 75 μ L |
| Detection: | Electrospray (negative ion), (SIR mode, m/z = 185) |
| Instrument: | Waters Alliance LC-MS with Micromass Platform |

LC Mass Detector

GC-MS or GC-FID Method

| | |
|-------------------|---|
| Column: | RTX 5 capillary, 30 meters, 0.25 mm ID, 0.25 μ m film thickness |
| Carrier gas: | Helium @ 30 cm/sec |
| Temp program: | 40 °C initial, 8 °C/ min to 300 °C |
| Injection volume: | 1 μ L |
| Detection: | HP 5972 MSD, (EI, SIM mode, m/z = 123) |

A) For LC-MS: No derivatization required. The MTBE* in the eluent is removed by evaporation and the extract is adjusted to a final volume of 1.0 mL with 10% methanol in water.

B) For GC: The eluent is heated for 40 min @ 60° C to convert endothall to the dimethyl ester. The ester is then extracted with DCM**. After removal of water by treatment with Na₂SO₄, the DCM** extract is evaporated to a final volume of 0.5 mL.

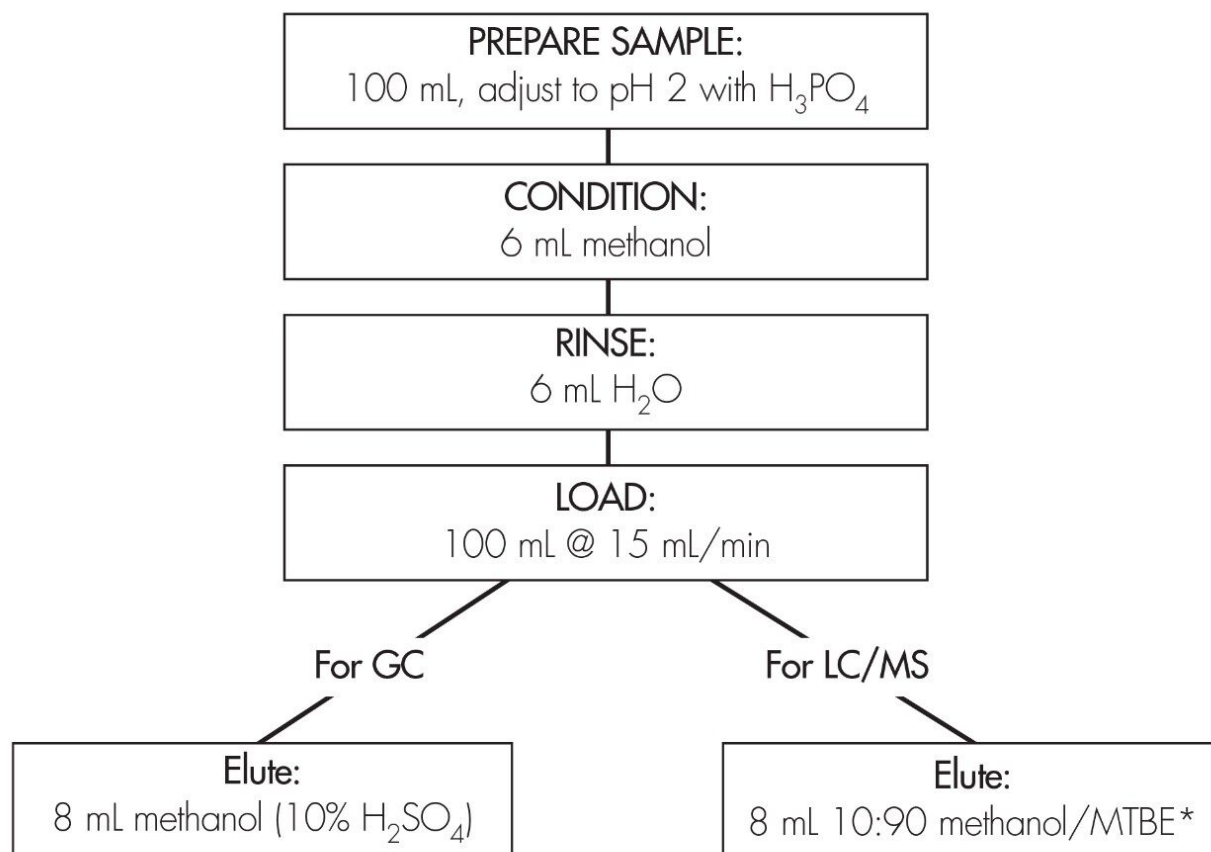
Soil Samples: The sample (10 g) is extracted with 35 mL pH 10 carbonate buffer (0.1M) followed by 20 mL of water. The combined extracts are adjusted to pH 2 with phosphoric acid and centrifuged. SPE is then performed using the same protocol as water samples.

* methyl butyl ether

** methylene chloride

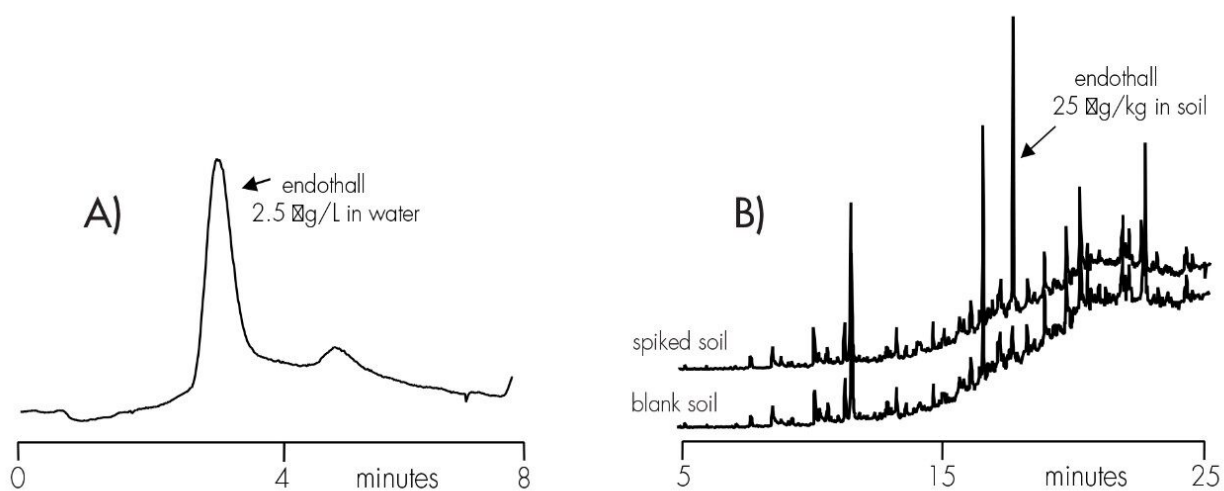
OASIS® HLB EXTRACTION METHOD

Conditions for Oasis® HLB Cartridge, 6 cc, 500 mg LP
Part Number 186000115



* methyl tbutyl ether
diethyl ether can be used as an alternative to MTBE

Results and Discussion



| % Recovery (% RSD) - LC/MS | | % Recovery (% RSD) - GC | |
|----------------------------|-----------------------|---------------------------|--------------------------|
| Tap water spike level | Tap water spike level | Soil (GC/FID) spike level | Soil (GC/MS) spike level |
| 2.5 µg/L | 10 µg/L | 100 µg/L | 25 µg/L |
| 4 replicates | 4 replicates | 4 replicates | 4 replicates |
| 81.1% (18%) | 99.6% (3.1%) | 81.8% (20%) | 76.2% (9.5%) |

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Alliance HPLC System <<https://www.waters.com/534293>>

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