Waters™

Application Note

Endothall in Drinking Water and Soil

Waters Corporation

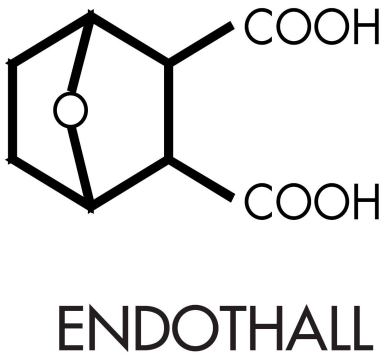
This is an Application Brief and does not contain a detailed Experimental section.

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 µm
Part number:	WAT058969
Mobile phase:	5% Acetonitrile in 1% formic acid/water
Flow rate:	200 µL/min
Injection volume:	75 µL
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 µm film thickness
Carrier gas:	Helium @ 30 cm/sec
Temp program:	40 °C initial, 8 °C/ min to 300 °C
Injection volume:	1 µL
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.

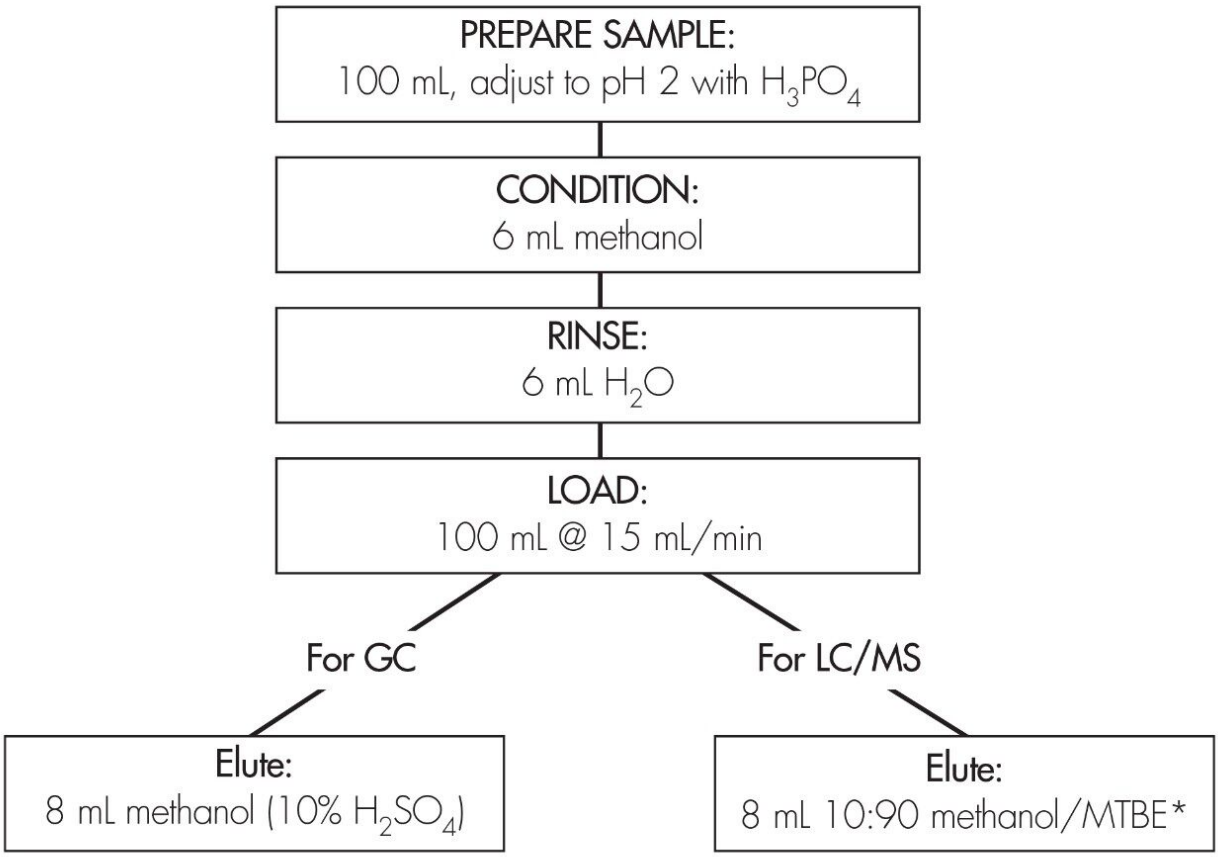
B) For GC: The eluent is heated for 40 min @ 60° C to convert endothall to the dimethyl ester. The ester is then extracted with DCM**. After removal of water by treatment with Na_2SO_4, the DCM** extract is evaporated to a final volume of 0.5 mL.

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

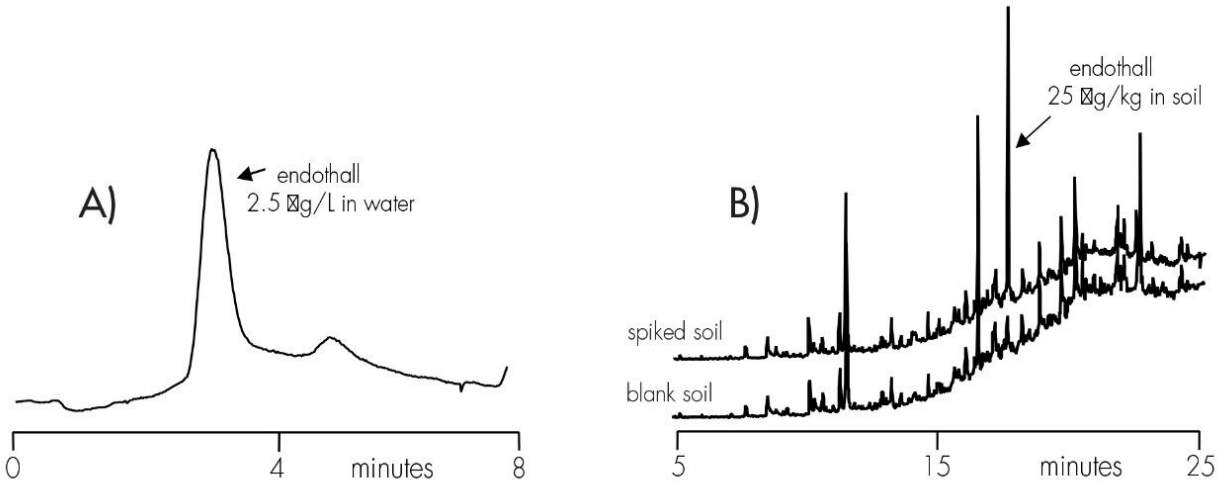
OASIS® HLB EXTRACTION METHOD

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



* methyl *t*-butyl 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%)

Featured Products

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

WA31764.77, June 2003