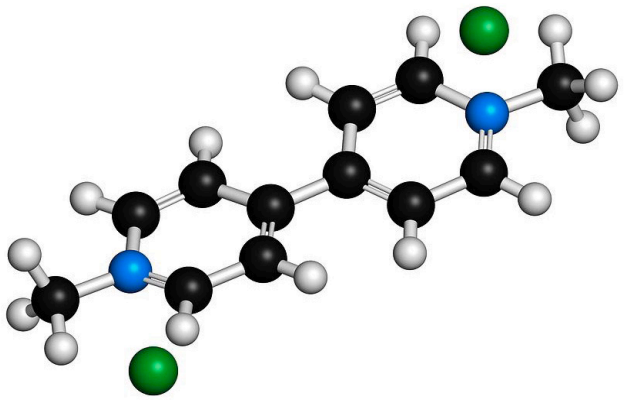
Determination of Endothall in Drinking Water by Ion-Exchange Extraction, Acid Methanol Methylation and Gas Chromatography/Mass Spectrometry



UCT Part Numbers

EC548006
Bio-Rex® 5, 6 mL

RFV0075P
75 mL empty reservoir

AD0000AS
Cartridge adapters

CLTTP050
Clean-Thru tips

Procedure:

1. Condition Cartridge

a) Remove the seal caps on each end of the **EC548006** cartridge, attach a Clean-Thru tip (to prevent corrosion of manifold lid caused by strong acid) to the bottom of the SPE cartridge, and a 75-mL reservoir to the top of the cartridge using a cartridge adaptor, and place on a vacuum manifold.

b) Draw each of the following reagents through the cartridge at a rate of 10 mL/minute, slow drip.

c) Add 10 mL of methanol and draw through to top of frit.

d) Add 10 mL of reagent water and draw through to top of frit.

e) Add 10 mL of 10% H_2SO_4 in methanol and draw through to top of frit.

f) Add 10 mL of reagent water and draw through to top of frit.

g) Add 10 mL of 0.1 N NaOH and draw through to top of frit.

h) Add 20 mL of reagent water and draw through to top of frit.

Note: Do not allow the cartridge to become dry between steps otherwise repeat steps starting with c)

It is critical that the extraction steps be followed exactly in order for the cartridge to effectively function in sample clean up and extraction.

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2. Sample Addition

a) Fill the reservoir with about 70 mL of sample (pH 5.5 to 7.5) and adjust vacuum for a sample flow rate of about 3 mL/minute.
b) Add the remaining sample to the reservoir.
c) After the sample has been drawn through the cartridge, rinse the sample bottle with 10 mL of methanol, apply the rinse to the reservoir and draw through.
d) Dry cartridge for 5 minutes under 10-20 in Hg vacuum.
e) Place a culture tube inside the manifold to collect the eluent.

3. Extract Elution

a) Elute the cartridge with 8 mL of 10% H_2SO_4 in methanol.
b) Follow with 6 mL of methylene chloride (CH_2Cl_2).
c) Elute over a 1 minute period.

4. Sample Derivatization and Partition

a) Place a cap on the culture tube and heat at 50 °C for 1 hour.
b) Pour the contents of the culture tube into a 125 mL separatory funnel rinsing the tube with 2 x 0.5 mL aliquots of methylene chloride.
c) Add the rinse to the separatory funnel.
d) Add 20 mL of 10% Na_2SO_4 in reagent water to the separatory funnel. Vigorously shake the separatory funnel several times venting the funnel each time.
e) Allow the phases to separate then drain the organic layer into a 15 mL graduated centrifuge tube.
f) Repeat the above extraction with 2 additional 2 mL aliquots of methylene chloride. Add this to the methylene chloride in the centrifuge tube, then concentrate to 1 mL.
g) Sample is ready for GC/MS analysis.

5. Analysis

a) Analyze the extract by injecting 1-2 µL of the concentrated extract into a GC/MS.
b) Identify endothall by comparison of its mass spectrum to a reference sample.

Retention Times and Method Detection Limits (MDL)

	Retention Time (min)		MDL (µg/L)		
Compound	**Column A**	**Column B**	**Column C**	**GC/MS**	**FID**
Endothall	16.02	19.85	18.32	1.79	0.7
Acenapththene-d10	14.69	-	-	-	-

Column A: DB-5 fused silica capillary for GC/MS, 30 m x 0.25 mm, 0.25 micron film | MS inlet temperature = 200°C | Injector temperature = 200°C | Temperature Program: Hold 5 minutes at 80°C, increase to 260°C at 10°/min, hold 10 minutes

Column B: FID primary column, RTX Volatiles, 30 m x 0.53 mm I.D., 2 micron film | Detector temperature = 280°C | Injector Temperature = 200°C | Carrier gas velocity = 50 cm/sec | Temperature program: Same as **Column A**.

Column C: FID confirmation column, DB-5, 30 m x 0.32 mm ID, 0.25 micron film. | Carrier Gas velocity = 27 cm/sec | Same injector, detector, and temperature program as **Column A.**

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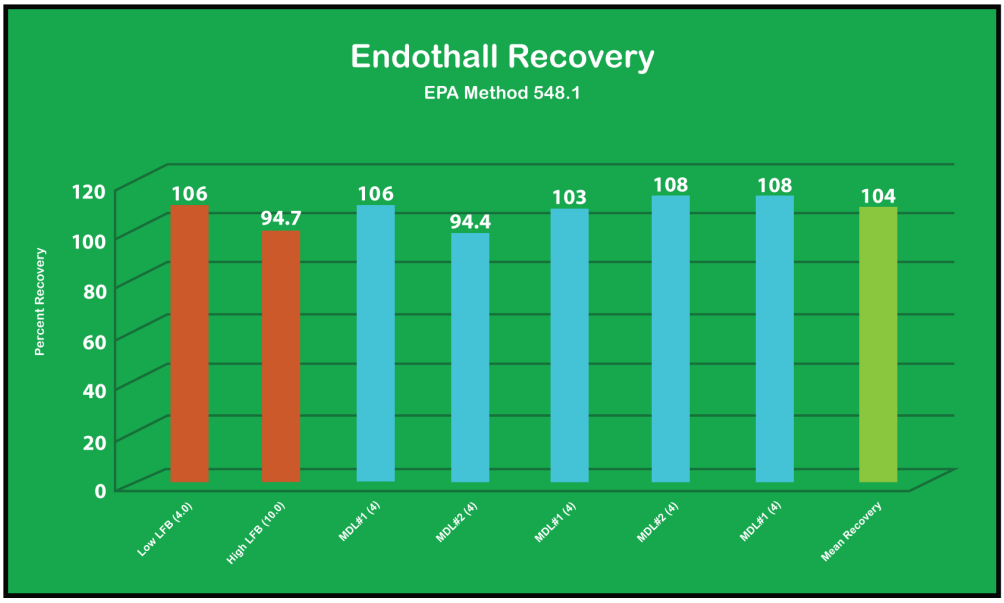

Endothall Recovery Using EC548006 Cartridges

Interferences

- Major potential interferences in this ion-exchange procedure are other naturally occurring ions such as calcium, magnesium and sulfate. Calcium and magnesium (>100 mg/L) can complex with the endothall anion and make it unavailable for capture as an anion.
- Sulfate anions (>250 mg/L) can act as a counter ion displacing anionic endothall on the ion exchange column. Elevated levels of these ions may contribute to reduced recovery of the primary analyte.

One or both of the following remedies may be used reduce these interferences:

- Sample dilution to reduce the concentration of these ions (10:1)
- Ethylenediamine tetraacetic acid (EDTA) addition to complex the cations (186 mg/100 mL sample)

For samples containing moderately high levels of these ions, add 186 mg of EDTA per 100 mL sample (0.005 M). For samples containing higher levels of sulfate, sample dilution may be required in addition to the EDTA.

Using western surface water as an example (2000 mg/L sulfate) it was successfully analyzed after dilution by a factor of 10 and the addition of 75 mg EDTA per 100 mL of the diluted sample (0.002 M). Samples containing intermediate levels of sulfate can be analyzed with smaller dilution factors. Guidelines on dilution factors and EDTA addition are shown below.

Sulfate mg/L	Dilution Factor	Added EDTA mg/100 mL
<250	1:1	186
250-500	1:2	125
500-1250	1:5	75
>1250	1:10	75

Note: Dilution should not be employed if adequate recovery is attained by the addition of EDTA alone

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References:

[1] *For complete details on Method 548.1 "Determination of Endothall in Drinking Water by Ion-Exchange Extraction, Acid Methanol Methylation and Gas Chromatography/Mass Spectrometry", the analyst is referred to: J. W. Hodgeson, August 1992, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

5108-13-01

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