Analysis of 1,4-Dioxane in 500 mL of Drinking Water by Gas Chromatography-Mass Spectrometry SIM Detection

Part Number: EU52112M6, 2 grams activated carbon in a 6 mL cartridge

1,4-dioxane (CAS 123-91-1), often referred to as dioxane, is a highly soluble non-biodegradable ether. This compound is used as solvent stabilizer to prevent the breakdown of chlorinated solvents.

1) Sample Preparation:

a) To dechlorinate the water sample add 25 mg of sodium sulfite, **then** acidify with 0.5 g of sodium bisulfate and swirl until dissolved.

2) Condition Cartridge:

a) Place a EU52112M6 cartridge on a single or multi-station vacuum manifold or automated extraction system
b) Add 3 mL of dichloromethane (methylene chloride) to the cartridge and draw through
c) Add 3 mL of methanol and draw through
d) Repeat step c)
e) Add 3 mL of DI water and draw through
f) Repeat step e) 4 times

3) Load Sample:

a) Add Internal standard * to the sample
b) Add sample to the cartridge. Adjust for a flow rate of 5 - 10 mL/minute
c) Dry cartridge by drawing air at full vacuum for 10 minutes

4) Elution:

a) Place a clean collection vial in the vacuum manifold
b) Add 3 mL of dichloromethane to the cartridge. Slowly draw through
c) Add a second 6 mL aliquot of dichloromethane and draw through
d) Add surrogate** (0.5 ppm) to the extract and bring to 10 mL final volume with dichloromethane
e) Dry extract by adding 2 g anhydrous sodium sulfate (Na_2SO_4) and vortex to mix the extract

5) Quantitate:

a) GC-MS-SIM (electron ionization)

6) Instrument & Conditions:

Column: Varian CP-Select 624 CB (6% cyanopropyl phenyl, 94& PDMS, 30 m 0.25mm x 1.4 um column) or equivalent
Injector: 200°C (Splitless mode)
Injector Volume: 1 µL
Flow: 1 mL minute
Oven: 30°C for 1 minute, 90°C at 7°C/minute, 200°C at 20°C/minute for 3 minutes

MS: quadrupole MS

SIM MODE

Dwell time 100 µs

Emission current 100 µA

***** Quantitation ions**

Segment 1 (0-8 minutes)	****THF-d_8**	**m/z 40***, 78, 80**
Segment 2 (8-16.5 minutes)	**1, 4 dioxane**	**m/z 58, 88*****
	***1, 4 dioxane-d_8**	**m/z 62, 64, 96*****

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*Grinmmett, Paul E., Munch, Jean W., Method Development for the Analysis of 1,4-Dioxane in Drinking Water Using Solid-Phase Extraction and Gas Chromatography-Mass Spectrometry, Journal of Chromatographic Science, Vol. 47, January 2009. Office of Research and Development, National Exposure Laboratory, Cincinnati, OH.

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