



EPA Method 522: DETERMINATION OF 1,4-DIOXANE IN DRINKING WATER BY SPE AND GC/MS WITH SELECTED ION MONITORING (SIM)*

UCT Part Numbers

EU52112M6

ENVIRO-CLEAN® 521 Coconut Carbon
2000mg/6mL Cartridge

VMF016GL

16 position glass block manifold



Summary:

1,4-dioxane (CASRN 123-91-1) referred to as dioxane, is a highly water soluble, non-biodegradable ether often times found as a trace contaminant of various chemicals used in cosmetics, detergents, and shampoos. Manufacturers now reduce 1,4-dioxane from these chemicals to low levels before they are incorporated into products used in the home. Prolonged exposure has been linked with damage to the central nervous system, liver and kidneys. The following detailed method utilizes a solid-phase extraction approach (SPE) coupled with gas chromatography/mass spectrometry (GC/MS) for the determination of this hazardous analyte in drinking water. Scan mode may be used if appropriate sensitivity is attained. This product can be used with most automated systems.

Procedure:

1. Sample Collection & Preservation

- a) Prepare bottles before sample collection with sodium sulfite (CASRN 7757-83-7).
- b) Open the tap and allow the system to flush until the water temperature has stabilized (approximately three to five min).
- c) Fill sample bottles. Do not flush out the dechlorination reagent.
- d) Cover the bottle and agitate by hand until the sodium sulfite has dissolved.
- e) Add sodium bisulfate (CASRN 7681-38-1). Mix until dissolved.

2. Sample Shipment and Storage

- a) Samples must be chilled during shipment and must not exceed 10° C during the first 48 hours after collection.
- b) Sample temperature must be confirmed to be at or below 10° C when they are received at the laboratory.
- c) Verification of sample dechlorination – Upon the receipt of samples at the laboratory, verify that field samples were dechlorinated at the time of collection. The absence of total chlorine can be verified with a N, N-DIETHYL-P-HENYLENEDIAMINE (DPD) CHLORINE TEST KIT – (Hach model CN-66; cat. # 2231-01 or equivalent).
- d) Holding time may be up to 28 days.



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3. Interferences

- a) All glassware must be meticulously cleaned with detergent and tap water, rinse with tap water, followed by reagent water.
- b) Non-volumetric glassware should be heated in a muffle furnace at 400° C for 2 hours.
- c) Volumetric glassware should be solvent rinsed with DCM or purge and trap grade methanol after washing, and dried in a low temperature oven (<120 °C) or air dried.
- d) Interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware. All items must be routinely demonstrated to be free from interferences (less than 1/3 the MRL for the method analyte) under the conditions of the analysis. Subtracting blank values from sample results is not permitted.
- e) Purge and trap grade methanol must be used for all steps where methanol is required. Other grades of methanol may contain low molecular weight compounds which may prohibit accurate identification and quantitation of the analyte, SUR and IS.
- f) Matrix interferences may be caused by contaminants that are co-extracted from the sample. The matrix interferences will vary considerably from source to source.
- g) Analyte carry-over may occur when a relatively “clean” sample is analyzed immediately after a sample containing relatively high concentrations of compounds.
- h) Syringes and splitless injection port liners must be cleaned carefully and replaced as needed. After analysis of a sample containing high concentrations of compounds, a LRB should be analyzed to ensure that accurate values are obtained for the next sample.
- i) During automated GC/MS analyses, extracts with positive results that were analyzed immediately following a sample with high concentrations of the analyte, should be reanalyzed after analyzing an acceptable LRB. If the analyte is not detected in extracts analyzed immediately after a high concentration extract, no reanalysis is necessary.
- j) Many silicone compounds may be leached from punctured autosampler vial septa, particularly when particles of the septa sit in the vial. This can occur after repeated injections from the same autosampler vial. If this method is performed in full scan mode, silicone compounds appear as regular chromatographic peaks with similar fragmentation patterns. They can unnecessarily complicate the total ion chromatogram.

4. Safety

- a) The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined.
- b) 1,4-Dioxane is classified as a class B2 or probable human carcinogen.
- c) Sodium bisulfate is used as a sample preservative to inhibit microbial growth and decay of 1,4-dioxane.

5. Sample Preparation Procedure - 100-500 mL samples

- a) Prepare Cartridge (vacuum manifold use).
- b) Place a EU52112M6 cartridge on a single or multi-station vacuum manifold.
- c) Add 3 mL of dichloromethane (methylene chloride) to the cartridge and draw through.
- d) Add 3 mL of methanol and draw through completely.
- e) Add 3 mL of DI water and draw through. Do not let cartridge go dry.
- f) Repeat step e) 5 times without letting the cartridge go dry.

Extract Sample

- g) Add surrogate 1,4-dioxane-d8 to the 100-500 mL sample water.
- h) Add sample water to the cartridge. Adjust vacuum for a 10 mL/minute flow rate.
- i) After the entire sample has drawn through, dry cartridge by drawing air at full vacuum. Cartridge will feel room temperature when dry.

Cartridge Elution

- j) Place a clean collection vial in the vacuum manifold.
- k) Fill each cartridge with dichloromethane (DCM). Soak sorbent for 1 minute then slowly draw through dropwise.



- l) Continue to add DCM to the cartridge until the total volume is 9 mL.
- m) Remove collection tube and adjust volume to 10 mL with DCM.
- n) Add IS. Adding 5 µl of 1000 µg/mL of IS solution to 10 mL DCM extract will yield 500 ng/mL.
- o) Mix by vortexing.
- p) Dry extract by adding 2 grams anhydrous sodium sulfate (Na₂SO₄).
- q) Mix well.
- r) Transfer aliquot to autosampler vials for analysis.

NOTE: This extract cannot be reliably concentrated by nitrogen evaporation because of the volatility of dioxane.

Instrument & Conditions:

Column: Varian CP-Select 624 CB (6% cyanopropyl phenyl, 94% PDMS, 30 m 0.25mm x 1.4 µm column (or equivalent))

Injector: 200°C (Splitless mode)

Injection Volume: 1 µL

Helium: Carrier gas 99.999% or better

Flow Rate: 1 mL/minute

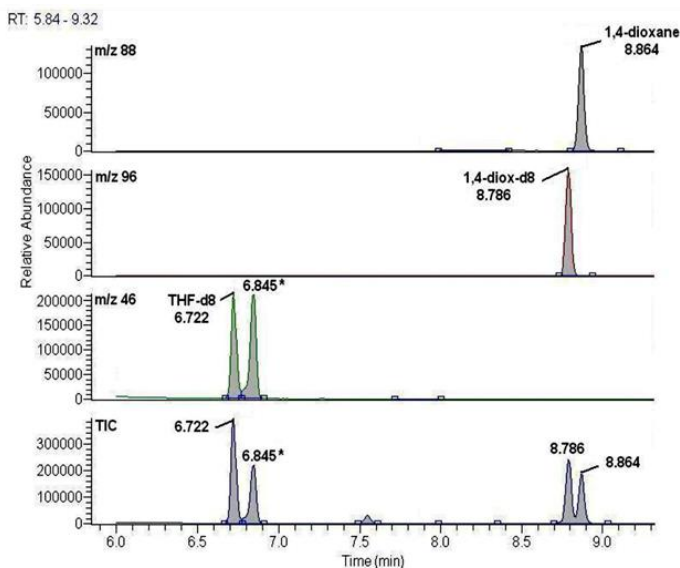
Temperature program: 30° C for 1 minute 90°C at 8° C/minute - 200° C at 20° C/minute for 4 minutes

MS: Any type of mass spectrometer may be used (i.e., quadrupole, ion trap, time of flight, etc.), although the SIM option may not provide enhanced sensitivity, or be an available option on all instruments

Dwell Time: 100 µs

Emission Current: 100 µA

Retention Times and Quantitation Ions (QIs)		
Analyte	RT	SIM Ions
1,4-dioxane	8.85	58,88
1,4-dioxane-d8	8.77	62, 64, 96
THF-d8 (IS)	6.68	46, 78, 80



Reconstructed total ion current chromatogram and mass chromatograms for THF-d8 (IS), 1,4-dioxane-d8 (SUR), and 1,4-dioxane at 0.5 mg/mL each (the standard is equivalent to an extract of a 10 mg/L aqueous sample). * Peak at 6.845 min is chloroform, a chemical present in DCM.

*Summarized from Jean W. Munch and Paul E. Grimmitt, "Method 522 Determination Of 1,4-Dioxane In Drinking Water By Solid Phase Extraction (SPE) And Gas Chromatography/Mass Spectrometry (GC/MS) With Selected Ion Monitoring (SIM)", Office of Research and Development, National Exposure Laboratory, Cincinnati, OH Version 1.0, September 2008

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