

1,4-DIOXANE IN WATER BY SELECTIVE ION MONITORING (SIM) GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
EPA CLP Method OLM03.1 (1994) ^a

Table 1A. Summary of Holding Times and Preservation for 1-4-Dioxane in Water by Selective Ion Monitoring (SIM) Gas Chromatography/Mass Spectrometry (GC/MS)

Analytical Parameter	Technical and Contract Holding Times	Preservation
1,4-Dioxane in Water	<u>Technical</u> : 14 days from collection to extraction and analysis; <u>Contract</u> : 10 days from receipt at laboratory to extraction and analysis	Cool to 4°C ±2°C

^a Analyze water samples for 1,4-dioxane following protocols for GC/MS analysis of volatile organic compounds (VOCs) outlined in the Contract Laboratory Program (CLP) Statement of Work (SOW) OLM03.1 with the following modifications:

1. Perform GC/MS analysis using selective ion monitoring rather than full scan analysis. Monitor ions of m/e 88 ±0.5 and 58 ±0.5 for 1,4-dioxane. Monitor ions of m/e 96 ±0.5 and 64 ±0.5 for internal standard 1,4-dioxane-d₈.
2. Use 25 mL aliquots of all standards, samples, and blanks.
3. Use a heated purge (50°C).

Data Calculations and Reporting Units:

Calculate the sample results using the procedure outlined in Section 11.2 of the CLP SOW OLM03.1.

Report water sample results in concentration units of micrograms per liter (µg/L).

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Compound List, CAS Number, and Contract Required Quantitation Limit for 1-4-Dioxane in Water by SIM GC/MS

COMPOUND	CAS No.	CRQL (µg/L)
1-4, Dioxane	123-91-1	5

Table 2. Summary of Calibration Procedures for 1-4-Dioxane in Water by SIM GC/MS

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
GC/MS Tuning with BFB	Beginning of each 12 hour period during which standards samples are analyzed	Ion abundance criteria provided in Table 1 of the VOA method in OLM03.1	1. Identify the problem. 2. MS tune criteria must be met before any calibration standards, samples, blanks, or QC samples are analyzed
Initial Calibration (minimum blank + 5 points) (ICAL) ^{a, b}	Initially; whenever required, due to failure of CCV	%RSD for RFs #30%	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at 10 µg/L (Separate source from ICAL standards)	Beginning of each 12-hour time period, after every 10 samples, and end of run	%D between RFs from CCV and average of ICAL RFs must be <±25%	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV

^a The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio \$5:1. If this requirement cannot be met, the laboratory must submit a method detection limit (MDL) study as part of the data package.

^b Prepare calibration standards containing 1,4-dioxane at 4, 10, 20, 30, and 40 ng/L in 25 mL of purge volume.

Table 3. Summary of Internal Quality Control Procedures for 1-4-Dioxane in Water by SIM GC/MS

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG ^a (1 per 20 samples minimum)	< CRQL	1. Investigate source of contamination and document 2. All samples processed with a method blank that is out of control must be re-analyzed
Instrument Blank	Following any analysis that exceeds the calibration range	< CRQL	1. Instrument blank must indicate a concentration below the CRQL before analysis can continue
Internal Standard (IS) (1,4-dioxane-d ₈)	Every standard, sample, method blank, and QC sample at 10 µg/L	IS area within a factor of two of the IS area in the associated CCV (-50% to +100%); Retention time (RT) within ±0.5 minutes of the IS RT in the associated CCV	1. Investigate the system for malfunction; 2. Re-analyze all samples analyzed during a system malfunction
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG	50-125% of expected value; #30 RPD between MS and MSD	1. Report in Case Narrative

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^b Spike 1-4-dioxane into the MS/MSD samples at a concentration equivalent to the mid-point calibration standard.

Dilute and re-analyze samples with concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.