

**1,2-DIBROMOETHANE (EDB) and 1,2-DIBROMO-3-CHLOROPROPANE (DBCP) IN WATER BY
MICROEXTRACTION AND GAS CHROMATOGRAPHY**
EPA Method 504.1 (1993)

Table 1A. Summary of Holding Times and Preservation for 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-Chloropropane (DBCP) in Water by Microextraction and Gas Chromatography

Analytical Parameter	Technical and Contract Holding Times	Preservation
EDB and DBCP	<u>Technical</u> : 28 days from collection to extraction and analysis; <u>Contract</u> : 26 days from receipt at laboratory to extraction and analysis	Sodium thiosulfate; Cool to 4°C ±2°C;

Data Calculations and Reporting Units:

Calculate the sample results using the average calibration factors from the ICAL or from the linear regression curve.

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

TABLE 1B. Target Compound List, CAS Numbers, and Contract Required Quantitation Limits for 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-Chloropropane (DBCP) in Water by Microextraction and Gas Chromatography

<u>COMPOUND</u>	<u>CAS No.</u>	<u>CRQL (µg/L)</u>
1,2-Dibromoethane (EDB)	106-93-4	0.05
1,2-dibromo-3-chloropropane (DBCP)	96-12-8	0.05

Table 2. Summary of Calibration Procedures for 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-Chloropropane (DBCP) in Water by Microextraction and Gas Chromatography

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) ^{a, b, c}	Initially; whenever required, due to failure of CCV	RSD for CFs #20%; or Linear Regression with a correlation coefficient (r) ≥ 0.99	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL (Separate source from ICAL standards)	Beginning of each 12-hour time period, after every 10 samples, and end of run	%D between nominal and calculated amount CF for each compound must be $< \pm 15\%$	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Retention time evaluation for CCV standards	Each analysis of CCV standards	± 3 x the SD of the avg ICAL RT for each analyte	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 $\geq 5:1$. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

^b Report the retention time window for each analyte. Determine retention time windows as ± 3 x the standard deviation of the average initial calibration retention time for each analyte.

^c ICAL and continuing CAL standards must contain all surrogate compounds and target analytes listed in Table 1B.

Table 3. Summary of Internal Quality Control Procedures for 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-Chloropropane (DBCP) in Water by Microextraction and Gas Chromatography

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG ^a (1 per 20 samples minimum) per analytical instrument	< CRQL for each compound	1. Investigate source of contamination and document 2. All samples processed with a method blank that is out of control must be re-extracted and re-analyzed
Surrogate Spike	Every standard, sample and method blank at 10 times CRQL	65-125% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries 2. If re-analysis does not solve the problem, re-extract and re-analyze
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	75-115% of expected value; #15 RPD between MS and MSD	1. Report in Case Narrative
Laboratory Control Sample (LCS) ^b	One per batch or SDG	60-140% of expected value	1. Re-extract and re-analyze the LCS and all samples associated with the non-compliant LCS

^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 each target compound into the LFB at a concentration of 0.25 Fg/L.

Perform weekly MDL checks according to Section 9.4 of Method 504.1 and analyze a sample from an external source quarterly according to Section 9.5 of Method 504.1. Document outliers in the case narrative.

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.

Second column confirmation is required for all positive results. Confirmation must be performed on a column of a phase different from that used for quantitation. Confirmation analyses must meet all calibration criteria specified in Table 2 and blank acceptance criteria specified in Table 3.