Designation: D5316 – 98 (Reapproved 2024)

Standard Test Method for 1,2-Dibromoethane and 1,2-Dibromo-3-Chloropropane in Water by Microextraction and Gas Chromatography[1]

This standard is issued under the fixed designation D5316; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of 1,2-dibromoethane (commonly referred to as ethylene dibromide or EDB) and 1,2-dibromo-3-chloropropane (commonly referred to as DBCP) in water at a minimum detection level of 0.010 µg/L by liquid-liquid extraction combined with gas-liquid chromatography. This test method is applicable to the analysis of drinking waters and groundwaters. It is not recommended for wastewaters, due to the potential for interferences from high concentrations of other extractable organics. Similar information can be found in EPA Method 504.

1.2 This test method was used successfully with reagent water and groundwater. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Sections 6 and 9.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1066 Practice for Sampling Steam

D1129 Terminology Relating to Water

D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits (Withdrawn 2003)[3]

D1193 Specification for Reagent Water

D3370 Practices for Sampling Water from Flowing Process Streams

D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water (Withdrawn 2024)[3]

D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn 2002)[3]

D5789 Practice for Writing Quality Control Specifications for Standard Test Methods for Organic Constituents (Withdrawn 2002)[3]

2.2 *U.S. EPA Standards:*[4]

Method 504 Methods for the Determination of Organic Compounds in Drinking Water, Revision 2.0, 1989

Method 524 Measurement of Purgeable Organic Compounds in Drinking Water by Gas Chromatography/Mass Spectrometry

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

4. Summary of Test Method

4.1 This test method consists of microextraction of the sample followed by gas chromatographic analysis of the extract.

4.2 An aliquot of the sample is extracted with hexane. Two µL of the extract are then injected into a gas chromatograph equipped with a linearized electron capture detector for separation and analysis. Aqueous calibration standards are extracted and analyzed in an identical manner as the samples in order to compensate for possible extraction losses.

[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

4.3 The extraction and analysis time is 30 min to 50 min per sample, depending upon the analytical conditions chosen.

4.4 Confirmatory evidence can be obtained using a dissimilar column. When component concentrations are sufficiently high, Gas Chromatography/Mass Spectrometric (GC/MS) methods may be used for confirmation analysis. (See EPA Method 524.2.)

5. Significance and Use

5.1 This test method is useful for the analysis of drinking water and groundwaters. Other waters may be analyzed by this test method, see 1.2.

5.2 EDB and DBCP have been widely used as soil fumigants. EDB is also used as a lead scavenger in leaded gasolines. These compounds are very water soluble and are often found in groundwater and drinking water. Since they are highly toxic and are suspected carcinogens, there is concern about the potential health impact of even extremely low concentrations in potable water.

6. Interferences

6.1 Impurities contained in the extracting solvent usually account for the majority of the analytical problems. Solvent blanks should be analyzed on each new bottle of solvent before use. Indirect daily checks on the extracting solvent are obtained by monitoring the water blanks. Whenever an interference is noted in the water blank, the analyst should reanalyze the extracting solvent. Low-level interferences generally can be removed by distillation or column chromatography.

NOTE 1—When a solvent is purified, stabilizers put into the solvent by the manufacturer are removed, thus potentially making the solvent hazardous. Also, when a solvent is purified, preservatives put into the solvent by the manufacturer are removed, thus potentially making the shelf-life short. However, it is generally more economical to obtain a new source of solvent. Interference-free solvent is defined as a solvent containing less than 0.1 µg/L individual analyte interference. Protect interference-free solvents by storing them in an area known to be free of organochlorine solvents.

6.2 This liquid-liquid extraction technique efficiently extracts a wide boiling range of nonpolar organic compounds and, in addition, extracts polar organic components of the sample with varying efficiencies.

6.3 Current column technology suffers from the fact that EDB at low concentrations may be masked by very high levels of dibromochloromethane (DBCM), a common disinfection by-product of chlorinated drinking waters.

7. Apparatus and Equipment

7.1 *Gas Chromatography (GC) System:*

7.1.1 The GC system must be capable of temperature programming and should be equipped with a linearized electron capture detector and a capillary column splitless injector at 200 °C. Separate heated zones for the injector and detector components are recommended.

7.1.2 Two gas chromatography columns are recommended. Column A (7.1.3) is a highly efficient column that provides separations for EDB and DBCP without interferences from trihalomethanes. Column A should be used as the primary analytical column unless routinely occurring analytes are not adequately resolved. Column B (7.1.4) is recommended for use as a confirmatory column when GC/MS confirmation is not viable.[5] Retention times for EDB and DBCP on these columns are presented in Table 1.

TABLE 1 Chromatographic Conditions for 1,2-dibromethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP)

Analyte	Retention Time (min)		
	Column A	Column B	Column C
EDB	9.5	8.9	4.1
DBCP	17.3	15.0	12.8

7.1.3 *Column A*—A 0.32 mm ID by 30 m long fused silica capillary with dimethyl silicone mixed phase.[6] The linear velocity of the helium carrier gas should be about 25 cm/s at 100 °C. The column temperature is programmed to hold at 40 °C for 4 min, to increase to 190 °C at 8 °C/min, and hold at 190 °C for 25 min or until all expected compounds have eluted. (See Fig. 1 for a sample chromatogram.)

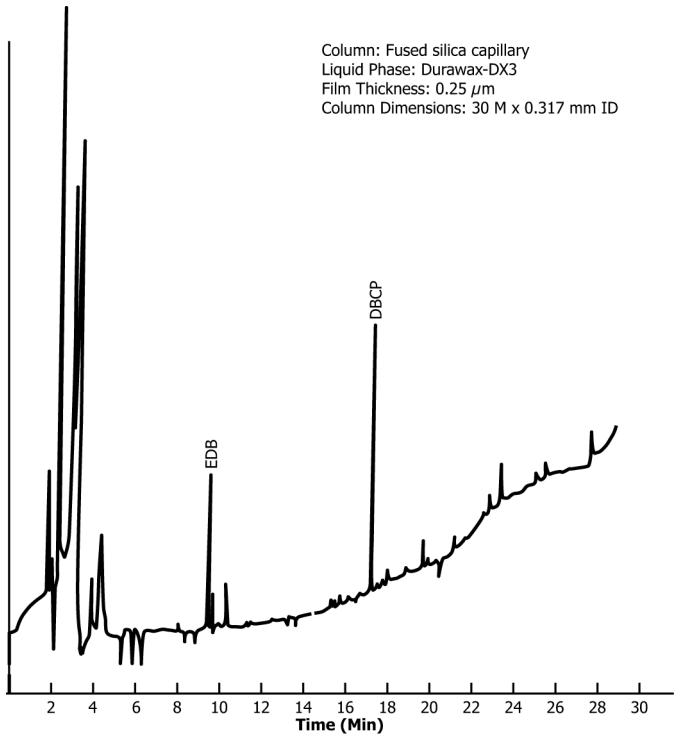

FIG. 1 Extract of Reagent Water Spiked at 0.114 µg/L with EDB and DBCP

7.1.4 *Column B (alternative column)*—A 0.32 mm ID by 30 m long fused silica capillary with methyl polysiloxane phase.[7] The linear velocity of the helium carrier gas should be

[5] An alternative column has been recommended by the Restek Corporation and is described in 7.1.5 as Column C.

[6] J & W Durawax DX-3, 0.25 µm, available from J & W Scientific, 91 Blue Ravine Rd., Folsom, CA 95630, or its equivalent, has been found suitable for this purpose.

[7] J & W DB-1, 1.0 µm film, available from J & W Scientific, or its equivalent, has been found suitable for this purpose.