Designation: D 3973 – 85 (Reapproved 2003)

Standard Test Method for Low-Molecular Weight Halogenated Hydrocarbons in Water[1]

This standard is issued under the fixed designation D 3973; 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 analysis of drinking water. It is also applicable to many environmental and waste waters when adequate validation is included.

1.2 This test method covers the determination of halomethanes, haloethanes, and some related extractable organohalides amenable to gas chromatographic measurement. The applicable concentration range for trihalomethanes is from 1 to 200 µg/L. Detection limits depend on the compound, matrix, and on the characteristics of the gas chromatographic system.

1.3 For compounds not specifically included in the precision and bias section the analyst should validate the test method by collecting precision and bias data on actual samples.

1.4 Confirmation of component identities is obtained by observing retention times using gas chromatographic columns of different polarities. When concentrations are sufficiently high (>50 µg/L) confirmation with halogen specific detectors or gas chromatography/mass spectrometry (GC/MS) may be used. Confirmation of purgeable compounds at levels down to 1 µg/L can be obtained using Test Method D 3871 with GC/MS detection.

1.5 *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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

D 3871 Test Method for Purgeable Organic Compounds in Water Using Headspace Sampling[3]

E 355 Practice for Gas Chromatography Terms and Relationships[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129 and Practice E 355.

4. Summary of Test Method

4.1 This test method employs liquid/liquid extraction to isolate compounds of interest and provide a five-fold concentration enhancement prior to measurement.[5,6,7]A5-mL aqueous sample is extracted once with 1 mL of solvent. -µL aliquot of the extract is analyzed in a gas chromatograph equipped with an electron-capture detector.

4.2 Extraction efficiencies with the 1:5 solvent/sample ratio for trihalomethanes average above 90 %. To compensate for extraction losses, calibration standards are extracted and analyzed in an identical manner.

4.3 The concentration of each component is calculated and reported in micrograms per litre.

5. Significance and Use

5.1 The incidental conversion of organic material to trihalomethanes and other volatile organohalides during chlorination of water is a possible health hazard and is the object of much research. This test method can be used as a rapid, simple means for determining many volatile organohalides in raw and processed water.

6. Interferences

6.1 Volatile compounds that are extractable and responsive to electron-capture detection may interfere with this test method.

6.2 Impurities in the extracting solvent can be a source of interference. Solvent blanks should be analyzed daily and before a new bottle of solvent is used for the first time.

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[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.

Current edition approved Jan. 10, 2003. Published January 2003. Originally approved in 1980. Last previous edition approved in 1995 as D 3973 – 85 (1995) ε1 .

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.02.

[4] *Annual Book of ASTM Standards*, Vol 14.01.

[5] Mieure, J. P., "A Rapid and Sensitive Method for Determining Volatile Organohalides in Water," *Journal AWWA*, Vol 69, 1977, p. 60.

[6] Richard, J. J., and Junk, G. A., "Liquid Extraction for Rapid Determination of Halomethanes in Water," *Journal AWWA* , Vol 69, 1977, p. 62.

[7] "The Analysis of Trihalomethanes in Drinking Water by Liquid/Liquid Extraction," U.S. Environmental Protection Agency, EMSL, Cincinnati, OH, Sept. 9, 1977.

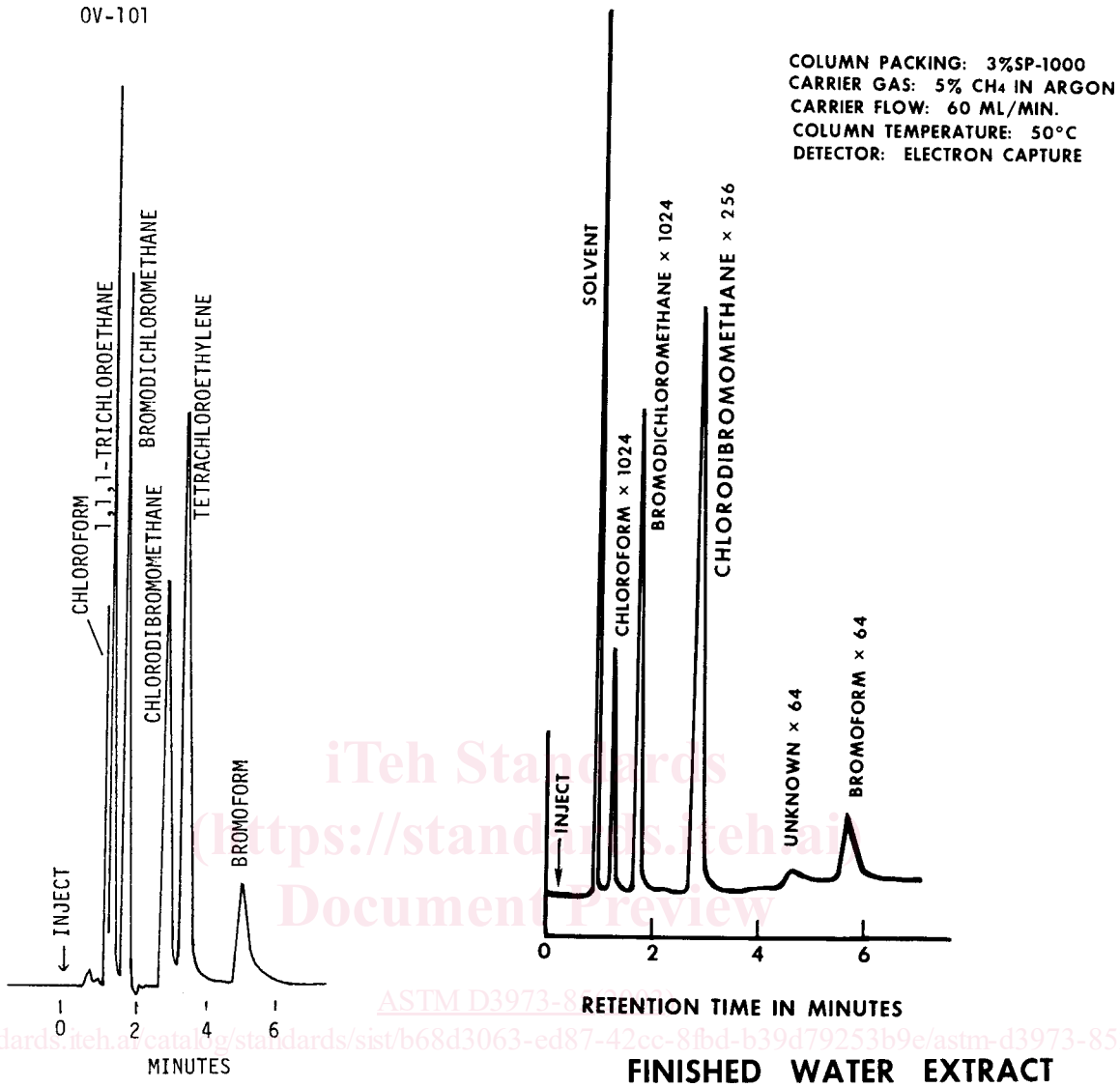

FIG. 1 Chromatograms of Standards

Whenever interfering compounds are traced to the solvent, a new source of solvent should be obtained. Alternatively, impurities may be removed by distillation, column chromatography[5] or purging with high-purity nitrogen or helium. This procedure is quantitative as long as solvent interference contributes less than 10 % to the component concentration in the sample.

7. Apparatus

7.1 *Extraction Vessel*, 9-mL (2-dram) vial with aluminum foil or PTFE-lined caps.

7.2 *Sample Containers*, 40-mL screw cap vials sealed with PTFE-faced silicone septa. [8]

7.3 *Micro Syringes*, 10, 100-µL.

7.4 *Pipets*, 1.0 and 5.0-mL transfer.

7.5 *Glass-Stoppered Volumetric Flasks*, 10 and 100-mL.

7.6 *Gas Chromatograph*, with electron-capture detector.

7.7 *Columns*, either of the following columns have been found suitable for this analysis. See Fig. 1 for chromatograms. If other column conditions are used, it is up to the analyst to demonstrate the precision and bias statements are met by collecting precision, bias, and recovery data.

7.7.1 *Nonpolar*, 3-mm inside diameter by 2-m long borosilicate glass packed with 100/120 mesh support [9] coated with a methyl silicone liquid phase [10] and operated at 60°C with 45 mL/min carrier flow.

7.7.2 *Polar*, 3-mm inside diameter by 2-m long borosilicate glass packed with 100/120 mesh support[9] coated with a polar

[8] 13075 vials and 12722 septa, available from Pierce Chemical Co., P.O. Box 117, Rockford, IL 61105, have been found suitable; other sources that are equivalent may be substituted.

[9] Gas-Chrom Q, available from Applied Science Laboratory, Inc., P.O. Box 440, State College, PA 16801, has been found suitable; other sources that are equivalent may be substituted.

[10] OV-101, available from Ohio Valley Specialty Chemical, Inc., Route 6, Marietta, OH 45750, has been found suitable; other sources that are equivalent may be substituted.