



Designation: D3695 – 95 (Reapproved 2021)^{ε1}

Standard Test Method for Volatile Alcohols in Water by Direct Aqueous-Injection Gas Chromatography¹

This standard is issued under the fixed designation D3695; 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.

^{ε1} NOTE—The WTO caveat was editorially added in November 2021.

1. Scope

1.1 This test method covers a wide range of alcohols with various structures and boiling points that can be separated and detected quantitatively in water and waste water at a minimum detection limit of approximately 1 mg/L by aqueous-injection gas-liquid chromatography.² This test method can also be used to detect other volatile organic compounds qualitatively. Organic acids, amines, and high boiling, highly polar compounds are not readily detectable under this set of conditions. For analysis of organics with similar functionalities, refer to other test methods in Volumes 11.01 and 11.02 of the *Annual Book of ASTM Standards*.

1.2 This test method utilizes the procedures and precautions as described in Practice D2908. Utilize the procedures and precautions as described therein.

1.3 This test method has been used successfully with reagent grade Type II and natural chlorinated tap waters. It is the user's responsibility to assure the validity of this test method for any untested matrices.

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

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *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:*³

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2908 Practice for Measuring Volatile Organic Matter in Water by Aqueous-Injection Gas Chromatography

D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water

D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn 2002)⁴

E355 Practice for Gas Chromatography Terms and Relationships

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129 and Practice E355.

4. Summary of Test Method

4.1 An aliquot of an aqueous sample is directly injected into a gas chromatograph by means of a microlitre syringe. The organic compounds in the sample are separated and eluted from a chromatographic column into a flame ionization detector. The compounds are identified by relative retention time or Kovats Index, and measured by direct comparison with corresponding standard responses.

5. Significance and Use

5.1 The major organic constituents in industrial waste water need to be identified for support of effective in-plant or

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 Sugar, J. W., and Conway, R. A., "Gas-Liquid Chromatographic Techniques for Petrochemical Waste Water Analysis," *Journal of the Water Pollution Control Federation*, Vol 40, 1968, pp. 1622–1631.

³ 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.

⁴ The last approved version of this historical standard is referenced on www.astm.org.

pollution control programs. Currently, the most practical means for tentatively identifying and measuring a range of volatile organic compounds is gas-liquid chromatography.

6. Interferences

6.1 Since the specified column and conditions are applicable to numerous organics, the possibility of one or more components having identical retention times is always present. Therefore, the analyst must determine the qualitative identity of the components of each peak by spectrometric techniques or a multi-column approach, or both, so that proper quantitation for those compounds of interest may be made. Refer to [Table 1](#) for relative retention data.

7. Apparatus

7.1 *Gas Chromatograph and Accessory Equipment*, described in Practice [D2908](#), Sections 7.1 through 7.6, is used for this analysis.

7.2 *Column*, Carbowax 20 M⁵ (5%) on 80/100 Acid Washed Chromosorb W,⁶ 6.1-m (20-ft), 3.2-mm (1/8-in.) in outside diameter, 0.508-mm (0.020-in.) wall thickness, stainless steel.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁷ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification [D1193](#), Type II.

8.3 *Calibration and Standardization*—Prepare a stock solution of the materials of interest by weighing a known amount of each, 1.00 g or less, diluting with water to 1 L, and mixing. Subsequent dilutions should be prepared as deemed necessary.

9. Sampling

9.1 Collect the sample in accordance with Section 9 of Practice [D2908](#).

⁵ The sole source of supply of the apparatus known to the committee at this time is Union Carbide Corp. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁶ The sole source of supply of the apparatus known to the committee at this time is Johns-Manville Products Co. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁷ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Kovats Index and Relative Retention Data for Typical Components^A

Component	Kovats Index (I _x)	Relative Retention ^B
Diethyl ether	580	0.17
<i>n</i> -Hexane	600	0.19
Isopropyl ether	600	0.19
Ethylene oxide	700	0.20
Acetaldehyde	700	0.20
Vinyl ethyl ether	700	0.20
<i>n</i> -Heptane	700	0.20
Propylene oxide	737	0.22
Vinyl isobutyl ether	796	0.26
Acetone	796	0.26
<i>n</i> -Butyl chloride	796	0.26
Cyclohexene	808	0.27
Acrolein	820	0.28
Methyl acetate	820	0.28
Vinyl <i>n</i> -butyl ether	833	0.29
Octene-1	842	0.30
<i>n</i> -Butyraldehyde	865	0.32
Vinyl acetate	887	0.34
Isopropyl acetate	887	0.34
Methyl ethyl ketone	908	0.36
Ethyl acetate	912	0.37
Methanol	916	0.38
Isopropanol	935	0.39
Dioxolane	943	0.40
Benzene	962	0.42
Ethyl acrylate	978	0.44
Isopropenyl acetate	983	0.45
Methyl <i>n</i> -propyl ketone	983	0.45
Methyl vinyl acetate	992	0.46
Ethanol	1000	0.47
Acrylonitrile	1007	0.48
Propyl acetate	1007	0.48
2-Methylpentanaldehyde	1026	0.51
<i>n</i> -Butyl ether	1026	0.51
Methyl isobutyl ketone	1035	0.52
Isobutyl acetate	1035	0.52
2-Ethylbutyraldehyde	1042	0.53
Acetonitrile	1050	0.54
1,2-Dichloropropane	1056	0.55
sec-Butyl alcohol	1056	0.55
Propylene dichloride	1065	0.57
2,3-Pentanedione	1080	0.60
Toluene	1080	0.60
<i>n</i> -Butyl acetate	1080	0.60
Ethylene dichloride	1092	0.62
<i>n</i> -Propanol	1100	0.63
Crotonaldehyde	1110	0.65
Paraldehyde	1118	0.66
1,4-Dioxane	1118	0.66
Isobutanol	1137	0.70
Mesityl oxide	1137	0.70
<i>n</i> -Methylmorpholine	1142	0.72
Methyl amyl acetate	1150	0.73
2-Pentanol	1157	0.74
primary-Amyl acetate (Isomers)	1157–1185	0.74–0.82
<i>p</i> -Xylene	1160	0.75
Ethyl benzene	1160	0.75
Ethylidene acetone	1170	0.77
Methyl isoamyl ketone	1173	0.78
<i>n</i> -Butanol	1185	0.82