Designation: D5008 – 22

Standard Test Method for Ethyl Methyl Pentanol Content and Purity Value of 2-Ethylhexanol By Gas Chromatography[1]

This standard is issued under the fixed designation D5008; 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 ethyl methyl pentanol content and purity value of 2-ethylhexanol.

1.2 Water and acid cannot be determined by this test method and must be determined in accordance with Test Methods D1613 and E203 and those results used to normalize the chromatographic data.

1.3 For purposes of determining conformance of an observed or a calculated value using this test method to relevant specifications, test result(s) shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E29.

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 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

1.6 *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 a specific hazard statement, see 6.1.1.

1.7 *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]

D1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E203 Test Method for Water Using Volumetric Karl Fischer Titration

3. Summary of Test Method

3.1 A representative specimen is introduced onto a capillary column. The 2-ethylhexanol is separated from the ethyl methyl pentanol and other impurities while the components are transported through the column by an inert carrier gas. The separated components are measured in the effluent by a flame ionization detector and the areas for the peaks are determined by a suitable integration technique. The data are interpreted by applying component detector response factors to the peak areas, and the relative concentrations are determined by relating the individual peak responses to the total peak response. Acidity and water are measured by Test Methods D1613 and E203, respectively, and the results are used to normalize the values obtained by gas chromatography. An internal standard procedure is also included as an alternative calculation technique. With this procedure, all impurities are determined relative to the internal standard and the purity value is determined by subtracting the sum of the impurities, water, and acid from 100.

4. Significance and Use

4.1 This test method is used to determine the purity value and ethyl methyl pentanol content of 2-ethylhexanol.

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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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.

5. Apparatus

5.1 *Chromatograph*—Any gas chromatograph designed or modified for use with capillary or wide-bore capillary columns. The gas chromatograph should be equipped with a flame ionization detector or other detector capable of operating with these columns and capable of detecting impurities at a level of 0.01 weight % with a signal to noise ratio of at least 5:1.

5.2 *Column*—Any column capable of resolving 2-ethylhexanol from ethyl methyl pentanol and other impurities that may be present. The peaks should be resolved quantitatively within a practical elapsed time. Columns that meet the requirement of this test method are listed in Table 1. Other columns may be used, provided the user establishes that a column gives the required separations.

5.3 *Specimen Introduction System*—Any system capable of introducing a representative specimen into the gas chromatograph may be used. A 1-µL syringe has been used successfully.

5.4 *Computing Integrator*—Any computing integrator capable of accurately determining the peak areas generated during this analysis.

5.5 *Analytical Balance*—The internal standard technique requires an analytical balance capable of measuring 0.1 mg.

6. Reagents and Materials

6.1 *Carrier Gas*—Helium, purified nitrogen, or hydrogen are suitable. The carrier gas should have a minimum purity of 99.95 mol %.

6.1.1 **Warning—**If hydrogen is used, take special safety precautions to ensure that the chromatographic system is free from leaks.

6.2 *Detector Gases*—Hydrogen and air are used for the flame ionization detector. If a make-up gas is used, helium or nitrogen are suitable.

6.3 *Standards for Calibration and Identification*—Standard samples for all identifiable components present are needed for identification by retention time, and for calibration for quantitative measurements. In the case of the internal standard method, pure (99.0 + %) 2-ethyl-1-butanol is specified as the internal standard. Any other internal standard may be used provided it is not present in the sample and doesn't interfere with any other chromatographic peak with the column used.

7. Calibration and Standardization

7.1 *Identification*—Select the conditions of column, column temperature and carrier-gas flow that will give the necessary component resolution (see Table 1). Determine the retention time for each component by injecting small amounts of the compound either separately or in mixtures.

7.2 *Standardization*—The area under each peak generated is considered a quantitative measure of the corresponding compound. The relative area is proportional to concentration if the detector responds equally to all the sample components. The response to different components is generally significantly different for flame ionization detectors. This difference in detector response may be corrected by use of relative response factors obtained by injecting and measuring the response of known blends. Using pure materials, prepare a calibration mixture with each component present in the appropriate amount. If an internal standard calculation technique is used, include the internal standard in this calibration mixture. If pure components are not available and interfering components are

TABLE 1 Conditions and Retention Times

	Case I	Case II	Case III
Column:			
Material	fused silica	fused silica	fused silica
Length, m	10	10	30
Inside diameter, mm	0.53	0.53	0.32
Liquid phase	immobilized polydimethylsiloxane	immobilized polydimethylsiloxane	immobilized polyethylene glycol
Film thickness,µ m	5	5	0.25
Injection system:	direct flash vaporization	direct flash vaporization	split
Injection specimen size, µ L	0.1	0.1	1
Temperatures:			
Column temperature, °C (isothermal)	85	85	120
Injection port temperature, °C	200	200	220
Detector temperature, °C	200	200	220
Gases:			
Carrier gas	helium	helium	helium
Carrier gas flow rate, mL/min	4	4	0.6
Carrier gas velocity, cm/s	30	30	20
Hydrogen flow rate (detector), mL/min	30	30	30
Air flow rate (detector), mL/min	300	300	300
Make-up gas	none	none	helium
Make-up flow rate (detector), mL/min	none	none	30
Injection split ratio	[A]	[A]	50:1
Calculation technique:	normalization	internal standard	normalization
Typical retention time, min:			
2-ethyl-2-butanol (internal standard)	[A]	2.24	[A]
Ethyl methyl pentanol	5.75	5.75	5.74
2-ethylhexanol	7.76	7.76	7.24

[A] Not applicable.