Designation: D5008 – 07 (Reapproved 2012)

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 *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.* For a specific hazard statement, see 6.1.1.

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

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.

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.

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[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.

***A Summary of Changes section appears at the end of this standard**

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.

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 present, then appropriate adjustments must be made in calculating the weights of the components present.

7.2.1 *Chromatographic Conditions*—Using a suitable method selected from Table 1, analyze a representative specimen of the calibration mixture.

7.2.2 If a computing integrator is used, follow the manufacturer's instruction manual to calculate relative response factors. If manual calculations are used, calculate relative response factors for each component as follows:

$$R = (A \times B)/(C \times D) \quad (1)$$

where:

R = response factor for component of interest,
A = peak area of reference component,
B = weight of component of interest in calibration mixture, g,
C = peak area for component of interest in calibration mixture, and
D = weight of reference component in calibration mixture, g.

NOTE 1—If a normalization calculation technique is used, 2-ethylhexanol will be the reference component. If an internal standard calculation technique is used, the internal standard will be the reference component.

7.2.3 The calibration factor generated for ethyl methyl pentanol can be used to calculate the concentration of any unknowns present.

8. Procedure

8.1 *Normalization Technique:*

8.1.1 Introduce a representative specimen into the chromatograph.

8.1.2 Using the same conditions as for calibration and standardization, determine the areas for all peaks.

8.2 *Internal Standard Technique:*

8.2.1 Weigh 0.1 g of internal standard into a vial. Record the weight to the nearest 0.1 mg.

8.2.2 Into the same vial, weigh 20.0 g of a representative specimen. Record the weight to the nearest 0.1 mg. Close the vial tightly and mix well.

8.2.3 Introduce a representative specimen of the mixture in 8.2.2 into the gas chromatograph.

8.2.4 Using the same conditions as for calibration and standardization, determine the areas for all peaks.

9. Calculation

9.1 *Normalization Technique:*

9.1.1 If a computing integrator is used, follow the manufacturer's instruction manual to compute the percent of each component of interest.

9.1.2 If manual calculations are made, apply the appropriate detector response factor to each peak to obtain the corrected peak response. Calculate the weight percent, W, of each component of interest as follows:

$$W = (E/F) \times (100 - G) \quad (2)$$

where:

E = corrected peak response for component of interest,
F = sum of corrected peak responses for all components, and
G = sum of water and acid as determined by Test Methods D1613 and E203.

9.2 *Internal Standard Technique :*

9.2.1 If a computing integrator is used, follow the manufacturer's instruction manual to compute the percent of each component except the 2-ethylhexanol.

9.2.2 If manual calculations are made, apply the appropriate detector response factor to each peak to obtain the corrected peak response. Calculate the weight percent W of each component, except the 2-ethylhexanol, as follows:

$$W = (H \times I \times 100)/(J \times K) \quad (3)$$

where:

H = corrected peak response for component of interest,
I = weight of internal standard added to sample, g,
J = corrected peak response for internal standard, and
K = weight of sample mixed with internal standard, g.

9.2.3 Calculate the purity value, P, in weight percent as shown below:

$$P = 100 - L \quad (4)$$

where:

L = sum of water, acid, and the weight percent of all components except 2-ethylhexanol.

10. Report

10.1 Report the following information:

10.1.1 The ethyl methyl pentanol content to the nearest 0.001 weight % and

10.1.2 The purity value to the nearest 0.01 weight %.

11. Precision and Bias[3]

11.1 In an interlaboratory study of this test method in which one operator in ten laboratories tested 2-ethylhexanol with a purity value of 99.653 and which contained 0.203 weight % of ethyl methyl pentanol, the within-laboratory standard deviation and between-laboratory standard deviation, with 9 df, respectively, were as follows:

	Standard Deviation, weight %	
	Within Laboratory	Between Laboratory
Ethyl methyl pentanol	0.002	0.011
2-Ethylhexanol	0.006	0.040

11.1.1 Based upon these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

11.1.2 *Repeatability*—Two results, each the mean of duplicates obtained by the same operator on different days, should be considered suspect if they differ by more that 0.02 weight % absolute for purity values or 0.007 weight % absolute for ethyl methyl pentanol.

11.1.3 *Reproducibility*—Two results, each the mean of duplicates obtained by operators in different laboratories, should be considered suspect if they differ by more than 0.13 weight % absolute for purity values or 0.036 weight % absolute for ethyl methyl pentanol.

11.2 *Bias*—The bias of this test method has not been determined because a suitable reference standard is not available.

12. Keywords

12.1 ethyl methyl pentanol content/composition by gas chromatography; purity by gas chromatography; 2-ethylhexanol

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[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1118.

SUMMARY OF CHANGES

Committee D01.35 has identified the location of selected changes to this standard since the last issue (D5008 - 01) that may impact the use of this standard. (Approved June 1, 2007.)

(1) Added reference to Practice E29 in the Scope section.

(2) Added Practice E29 to list of Referenced Documents.

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