Designation: D7871 – 23

Standard Test Method for Analysis of Cyclohexane by Gas Chromatography (Effective Carbon Number)[1]

This standard is issued under the fixed designation D7871; 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 the purity of cyclohexane by gas chromatography.

1.2 This test method has been found applicable to the measurement of impurities such as those found in Table 1, which are impurities that may be found in cyclohexane. The impurities can be analyzed over a range with an upper limit of 400 mg/kg by this method, but may be applicable to a wider range. The limit of detection (LOD) is 0.7 mg/kg and the limit of quantitation (LOQ) is 2.3 mg/kg.

NOTE 1—The LOD and LOQ were calculated from the ILS data for benzene.

1.3 The following applies for the purposes of determining the conformance of the test results using this test method to applicable specifications: results shall be rounded off 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazards statements, see Section 8.

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:*[2]

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

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

E355 Practice for Gas Chromatography Terms and Relationships

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

2.2 *Other Standards:*[3]

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

3. Terminology

3.1 See Terminology D4790 for definitions of terms used in this test method.

4. Summary of Test Method

4.1 The specimen to be analyzed is injected into a gas chromatograph equipped with a flame ionization detector (FID) and a capillary column. The peak area of each component is measured and adjusted using effective carbon number (ECN)[4] correction factors. The concentration of each component is calculated based on its relative percentages of total adjusted peak area and normalized to 100.0000 %.

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.01 on Benzene, Toluene, Xylenes, Cyclohexane and Their Derivatives.

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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] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

[4] Scanlon, J. T. and Willis, D. E., "Calculation of Flame Ionization Detector Relative Response Factors Using the Effective Carbon Concept," *Journal of Chromatographic Science*, Vol. 23, August 1985, ppl. 333-339.

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

TABLE 1 Impurities Known or Suggested to be Present in Commercial Cyclohexane

C_4
(1) *n*-butane
(2) isobutene
C_5
(3) *n*-pentane
(4) isopentane
(5) cyclopentane
C_6
(6) *n*-hexane
(7) 2-methylpentane
(8) 3-methylpentane
(9) methylcyclopentane
(10) benzene
(11) cyclohexane
(12) 2,2-dimethylbutane
(13) 2,3-dimethylbutane
C_7
(14) 3,3-dimethylpentane
(15) 2,2-dimethylpentane
(16) 2,3-dimethylpentane
(17) 2,4-dimethylpentane
(18) 1,1-dimethylcyclopentane
(19) *trans*-1,3-dimethylcyclopentane
(20) *trans*-1,2-dimethylcyclopentane
(21) *cis*-1,2-dimethylcyclopentane
(22) 2,2-dimethylcyclopentane
(23) 2,4-dimethylcyclopentane
(24) *cis*-1,3-dimethylcyclopentane
(25) ethylcyclopentane
(26) methylcyclohexane
(27) 3-ethylpentane
(28) 3-methylhexane
(29) 2-methylhexane
(30) *n*-heptane
(31) toluene
C_8
(32) *iso*-octane
(33) *p*-xylene
C_9
(34) isopropylcylohexane

5. Significance and Use

5.1 This test method is suitable for setting specifications on the materials referenced in Table 1 and for use as an internal quality control tool where cyclohexane is produced or is used in a manufacturing process. It may also be used in development or research work involving cyclohexane.

5.2 This test method is useful in determining the purity of cyclohexane with normal impurities present. If extremely high boiling or unusual impurities are present in the cyclohexane, this test method would not necessarily detect them and the purity calculation would be erroneous.

TABLE 2 Recommended Operating Conditions

Detector	flame ionization
Injection Port	capillary splitter
Column A:	
Tubing	fused silica
Stationary phase	bonded and crosslinked 100 % dimethylpolysiloxane
Film thickness, µm	0.5
Length, m	100
Diameter, mm	0.25
Temperatures:	
Injector, °C	230
Detector, °C	250
Oven, °C	32 hold for 12 min Ramp 1 = 8 °C/min to 64 °C, hold for 10 min Ramp 2 = 10 °C/min to 200 °C, hold for 5 min
Carrier gas	Hydrogen
Flow rate, mL/min	3
Split ratio	100:1
Sample size, µl	1.0

6. Apparatus

6.1 *Gas Chromatograph*—Any instrument having a flame ionization detector that can be operated at the conditions given in Table 2. The system should have sufficient sensitivity to obtain a minimum peak height response for 1 mg/kg benzene of twice the height of the signal background noise.

6.2 *Columns*—The choice of column is based on resolution requirements. Any column may be used that is capable of resolving all significant impurities from cyclohexane. The column and conditions described in Table 2 have been used successfully and shall be used as a referee in cases of dispute.

6.3 Chromatographic data systems are preferred but electronic integration may be used if the user can demonstrate that the results are consistent with the precision statement.

6.4 *Injector*—An autoinjector is required.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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.[5] 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.

7.2 *Equipment Set-up Check Sample*:

7.2.1 Prepare peak identification material by pipetting the following amounts into a 250 mL flask:

10 mL isopentane CAS 78-78-4
5 mL *n*-pentane CAS 109-66-0
10 mL 2,2-dimethylbutane CAS 75-83-2
5 mL 2,3-dimethylbutane CAS 79-29-8
10 mL cyclopentane CAS 287-92-3
5 mL 2-methylpentane CAS 107-83-5
10 mL 3-methylpentane CAS 96-14-0
5 mL *n*-hexane CAS 110-54-3
10 mL 2,2-dimethylpentane CAS 590-35-2
5 mL methylcyclopentane CAS 96-37-7
10 mL 2,4-dimethylpentane CAS 108-08-7
1 mL benzene CAS 71-43-2
5 mL 2,3-dimethylpentane CAS 565-59-3
10 mL Iso-octane CAS 540-84-1

[5] *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.