Designation: D 7266 – 07

Standard Test Method for Analysis of Cyclohexane by Gas Chromatography (External Standard)[1]

This standard is issued under the fixed designation D 7266; 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. Calibration of the gas chromatography system is done by the external standard calibration technique.

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 of 5 to 180 mg/kg by this method, but may be applicable to a wider range.

1.3 The limit of detection is 1 mg/kg.

1.4 In 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 E 29.

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

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 3437 Practice for Sampling and Handling Liquid Cyclic Products

D 4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

D 6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E 355 Practice for Gas Chromatography Terms and Relationships

E 1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

2.2 *Other Document:*

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200 [3]

3. Terminology

3.1 See Terminology D 4790 for definitions of terms used in this test method.

4. Summary of Test Method

4.1 Cyclohexane is analyzed using a gas chromatograph (GC) equipped with a flame ionization detector (FID). A precisely repeatable volume of the sample to be analyzed is injected onto the gas chromatograph. The peak areas of the impurities are measured and converted to concentrations via an external standard methodology. Purity by GC (the cyclohexane content) is calculated by subtracting the sum of the impurities from 100.00. Individual impurities are reported in mg/kg. The cyclohexane purity is reported in weight percent.

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.

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[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons 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.

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

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

[A] These components were used to prepare the standard used in the repeatability program.

TABLE 2 Instrumental Parameters

Detector	flame ionization
Injection Port	capillary splitter
Column A:	
Tubing	fused silica
Stationary phase	bonded and crosslinked 100% dimethylpolysilicane
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, mls/min	3
Split ratio	100:1
Sample size, µl	1.0

TABLE 3 Summary of Precision Data (mg/kg)

Impurity	Expected Value	Average	Repeatability
Isopentane	21.1	21.1	2.0
n-pentane	24.7	25.8	3.0
2,2-dimethylbutane	9.9	9.9	1.0
cyclopentane	11.5	11.4	0.8
2,3-diemthylbutane	10.0	10.2	1.0
2-methylpentane	17.3	18.1	2.2
3-methylpentane	23.9	24.8	2.0
n-hexane	46.7	48.4	5.2
2,2-dimethylpentane	4.9	5.2	0.8
methycylopentane	36.1	36.8	2.1
2,4-dimethylpentane	49.7	51.7	5.1
benzene	12.1	12.4	1.3
2,3-dimethylpentane	57.3	58.3	4.2
1,1-dimethylcyclopentane	23.5	23.0	1.2
cyclohexene	29.4	29.6	1.3
3-methylhexane	9.9	10.5	0.9
cis-1,3-dimethylcyclopentane	22.6	23.6	1.6
trans-1,3-dimethylcyclopentane	10.8	11.1	0.8
3-ethylpentane	29.9	31.0	2.5
trans-1,2-dimethylcyclopentane	41.2	40.4	2.3
isooctane	10.0	10.5	1.1
n-heptane	37.1	38.4	3.7
methylcyclohexane	178.5	181.0	10.0
ethylcyclopentane	19.0	19.6	1.7
toluene	19.9	20.9	1.9
para-xylene	19.9	20.9	2.0
isopropylcyclohexane	19.6	20.4	1.8

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 described in Table 2 has been used successfully.

6.3 *Recorder*—Electronic integration is required.

6.4 *Injector*—The specimen must be precisely and repeatably injected into the gas chromatograph. An automatic sample injection device is highly recommended. Manual injection can be employed if the precision stated in Table 3 can be reliably and consistently satisfied.

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.[4] 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 *Gases*—Helium, hydrogen, nitrogen, or other as carrier. Carrier, makeup, and detector gases (except air) 99.999 % minimum purity. Oxygen in carrier gas less than 1 ppm, less than 0.5 ppm is preferred. Purify carrier, makeup, and detector gases to remove oxygen, water, and hydrocarbons. Purify air to

[4] *Reagent Chemicals, American Chemical Society Specifications* , 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.