Designation: ~~D7871 – 19~~ 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 ~~of 1 to~~ 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 limit of detection is 1 mg/kg.~~

1.3 ~~In~~ The following applies for the purposes of determining the conformance of the test results using this test method to applicable ~~specifications,~~ 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

---

[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.

Current edition approved ~~Nov. 1, 2019~~April 1, 2023. Published ~~November 2019~~April 2023. Originally approved in 2013. Last previous edition approved in ~~2018~~2019 as ~~D7871 – 18.~~D7871 – 19. DOI: ~~10.1520/D7871-19.~~10.1520/D7871-23.

[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 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

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 %.

---

[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.

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.

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 ~~has been used successfully.~~have been used successfully and shall be used as a referee in cases of dispute.

6.3 ~~*Recorder*—Electronic integration is required.~~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*—~~The specimen must be precisely and repeatably injected into the gas chromatograph. An automatic sample injection device is highly recommended. Manual injection may be employed if the precision stated in Tables 4–8 can be reliably and consistently satisfied.~~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

TABLE 2 ~~Instrumental Parameters~~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~~ 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~~
Flow rate, mL/min	3
Split ratio	100:1
Sample size, µl	1.0

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 mL *n*-heptane CAS 142-82-5
10 mL methylcyclohexane CAS 108-87-2
5 mL ethylcyclopentane CAS 1640-89-7
10 mL Toluene CAS108-88-3
5 mL *p*-xylene CAS 106-42-3
10 mL isopropylcylohexane CAS 696-29-7
5 mL 1,1-dimethylcyclopentane CAS 1638-26-2
10 mL *trans*-1,3-dimethylcyclopentane CAS 1759-58-6
5 mL *trans*-1,2 dimethylcyclopentane CAS 822-50-4
10 mL *cis*-1,2-dimethylcyclopentane CAS 1192-18-3
5 mL *cis*-1,3-dimethylcyclopentane CAS 2532-58-3
5 mL 3-ethylpentane CAS 617-78-7
10 mL 3-methylhexane CAS 589-34-4
5 mL 2-methylhexane CAS 591-76-4

7.2.1.1 Components not required for peak identification may be omitted.

7.2.1.2 Dilute to volume with high purity cyclohexane CAS 110-82-7.

7.2.2 Prepare sensitivity check material.

7.2.2.1 Partially fill a 500 mL volumetric flask with high purity cyclohexane.

7.2.2.2 Pipette 0.5 mL of *n*-octane (111-65-9) to the 500 mL volumetric flask.

7.2.2.3 Fill the 500 mL volumetric flask to the mark with high purity cyclohexane and mix thoroughly.

7.2.3 Preparation of the final equipment set-up check sample:

7.2.3.1 Partially fill a 500 mL volumetric flask with high purity cyclohexane.

7.2.3.2 Pipette 1 mL of the peak identification material prepared in 7.2.1 into the 500 mL volumetric flask

7.2.3.3 Pipette 1 mL of the sensitivity check material prepared in 7.2.2 into the 500 mL volumetric flask.

---

[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.

7.2.3.4 Dilute to volume with high purity cyclohexane and mix thoroughly.

7.2.4 The equipment set-up check sample may be purchased if available.

7.2.5 The purpose of the set-up check sample is to help determine the retention time of the various components and that the set-up has sensitivity specified by this standard. This sample shall not be used for calibration.

7.3 *Gases*—Helium, hydrogen, nitrogen, or other as carrier are permitted. Hydrogen carrier gas was used to develop this standard. Use of helium or nitrogen require different conditions. The user must conduct the necessary evaluation to determine that equivalent results are obtained. 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 remove hydrocarbons and water, and the air used for an FID should contain less than 0.1 ppm total hydrocarbons.

8. Hazards

8.1 Consult current OSHA regulations, suppliers' Safety Data Sheets, and local regulations for all materials listed in this test method.

9. Sampling and Handling

9.1 Sample the material in accordance with Practice D3437.

10. Preparation of Apparatus

10.1 Follow manufacturer's instructions for mounting and conditioning the column into the chromatograph and adjusting the instrument to the conditions described in Table 2 allowing sufficient time for the equipment to reach equilibrium. See Practices E1510 and E355 for additional information on gas chromatography practices and terminology.

~~11. Calibration and Standardization~~

~~11.1 Prior to implementation of the ECN method, a laboratory should demonstrate that acceptable precision and bias can be obtained using a synthetic mixture of known composition (calibration check sample). (Fig. 1)~~

11. Identification and Sensitivity Check

11.1 Prior to implementation of the ECN method, the laboratory should analyze the Equipment Set Up Check Sample to confirm retention times of the components and to determine adequate sensitivity (Fig. 1).

11.2 The LOD for this standard is 0.7 mg/kg. The equipment set-up check sample contains 1.8 mg/kg *n*-octane. Acceptable results are 0.9 mg/kg to 3 mg/kg.

11.3 The Equipment Set-up Check Sample should be analyzed as needed to confirm retention times and sensitivity.

12. Procedure

12.1 Bring the sample to room temperature.

12.2 Check the chromatography performance to make sure that the column is properly resolving peaks.

12.3 Inject into the gas chromatograph an appropriate amount of sample as previously determined in accordance with 6.1 and start the analysis.

12.4 Obtain a chromatogram and peak integration report.

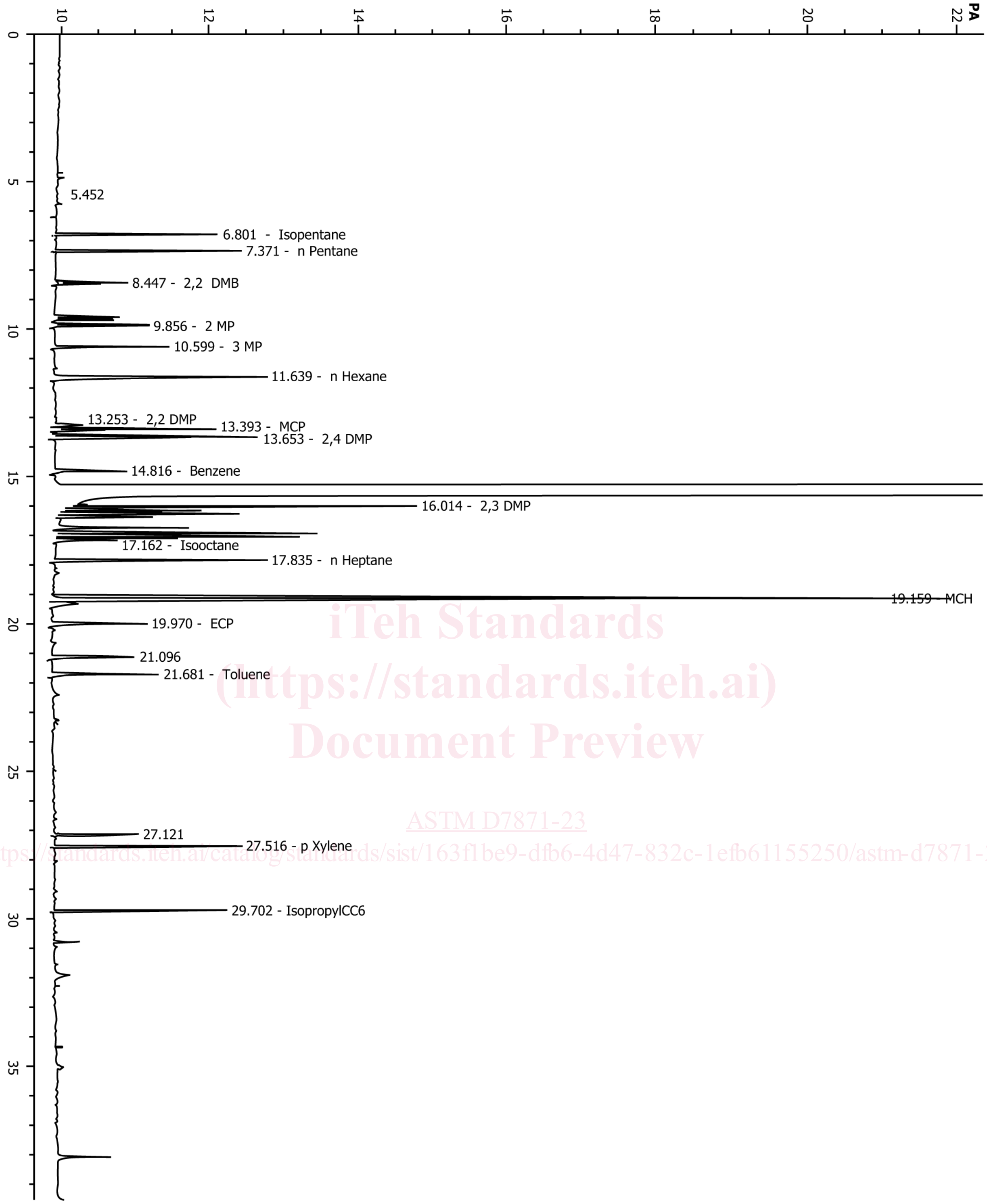

FIG. 1 Typical Chromatogram of Calibration Mixture Using Conditions in Table 2

TABLE 3 Effective Carbon Number Correction Factors

Component	Correction Factor (Mass)[A]
benzene	0.9095
butane	1.0151
cyclohexane	0.9799
cyclopentane	0.9799
2-methylpentane	1.0034
3-methylpentane	1.0034
2,2-dimethylpentane	1.0000
2,3-dimethylpentane	1.0000
2,4-dimethylpentane	1.0000
2,2-dimethylbutane	1.0034
2,3-dimethylbutane	1.0034
1,1-dimethylcyclopentane	0.9799
3-ethylpentane	0.9799
hexane	1.0034
methylcyclopentane	0.9799
2-methylhexane	1.0000
n-pentane	1.0080
toluene	0.9195
o-xylene	0.9271
m,p-xylene	0.9271
unidentified peaks	1.0000
n-octane	0.9975

[A] Mass correction factors are relative to *n*-heptane.

13. Calculations

13.1 Using the ECN mass correction factors listed in Table 3, calculate the concentration of each component as follows:

$$C_i = (A_i \times R_i) \times 1{,}000{,}000 \Sigma(A_i \times R_i) \quad (1)$$

where:

~~C_i = concentraion for component i, mg/kg,~~

C_i = concentration for component i, mg/kg,

A_i = peak area of component i, and

R_i = ECN mass correction factor for component i.

13.2 Calculate the purity of cyclohexane as follows:

$$\text{Cyclohexane, mass percent} = C_i/10{,}000 \quad (2)$$

where C_i is mg/kg of cyclohexane.

14. Report

14.1 Report the individual impurities to the nearest mg/kg.

14.2 Report the purity of cyclohexane to the nearest 0.01 mass %.

15. Precision and Bias[6]

15.1 An ILS was conducted which included two laboratories analyzing six ~~sample~~samples three times. Practice E691 was followed for the design and analysis of the data; this ILS did not meet Practice E691 minimum requirements of six labs, four materials, and two replicates. The details are given in ASTM Research Report: RR:D16-1046.

15.2 *Repeatability (r)*—Results should not be suspect unless they differ by more than shown in Tables 4-8. Results differing by less than "*r*" have a 95 % probability of being correct.

---

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1046. Contact ASTM Customer Service at service@astm.org.