



Designation: ~~D7266 – 07~~^{ε1} D7266 – 13

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

This standard is issued under the fixed designation D7266; 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.

^{ε1} NOTE—Table 2 was corrected editorially in July 2008.

1. Scope-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 ~~53~~ to ~~180~~200 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 [E29](#).

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

[D3437 Practice for Sampling and Handling Liquid Cyclic Products](#)

[D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards](#)

[D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals](#)

[D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials](#)³

[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 Document:*

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

3. Terminology

3.1 See Terminology [D4790](#) 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

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>.

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

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.

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** Tables 3–7 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 remove hydrocarbons and water, and the air used for an FID should contain less than 0.1 ppm total hydrocarbons.

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.

TABLE 2 Instrumental Parameters

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, $^{\circ}\text{C}$	230
—Detector, $^{\circ}\text{C}$	250
—Oven, $^{\circ}\text{C}$	32 hold for 12 min Ramp 1 = $8^{\circ}\text{C}/\text{min}$ to 64°C ; hold for 10 min Ramp 2 = $10^{\circ}\text{C}/\text{min}$ to 200°C , hold for 5 min
Carrier gas	Hydrogen
Flow rate, ml/min	3
Split ratio	100:1
Sample size, μl	1.0

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

TABLE 3 Summary of Precision Data (mg/kg)

Expected Value	Average	Repeatability
Isopentane	-21.1	-2.0
<i>n</i> -pentane	-24.7	-3.0
2,2-dimethylbutane	-9.9	-1.0
cyclopentane	-11.5	-0.8
2,3-dimethylbutane	-10.0	-1.0
2-methylpentane	-17.3	-2.2
3-methylpentane	-23.9	-2.0
<i>n</i> -hexane	-46.7	-5.2
2,2-dimethylpentane	-4.9	-0.8
methylcyclopentane	-36.1	-2.1
2,4-dimethylpentane	-49.7	-5.1
benzene	-12.1	-1.3
2,3-dimethylpentane	-57.3	-4.2
1,1-dimethylcyclopentane	-23.5	-1.2
cyclohexene	-29.4	-1.3
3-methylhexane	-9.9	-0.9
<i>cis</i> -1,3-dimethylcyclopentane	-22.6	-1.6
<i>trans</i> -1,3-dimethylcyclopentane	-10.8	-0.8
3-ethylpentane	-29.9	-2.5
<i>trans</i> -1,2-dimethylcyclopentane	-41.2	-2.3
isooctane	-10.0	-1.1
<i>n</i> -heptane	-37.1	-3.7
methylcyclohexane	178.5	10.0
ethylcyclopentane	-19.0	-1.7
toluene	-19.9	-1.9
<i>para</i> -xylene	-19.9	-2.0
isopropylcyclohexane	-19.6	-1.8

TABLE 3 Methylcyclopentane (mg/kg)

	Average \bar{X}	Repeatability Limit r	Reproducibility Limit R
Blank	93	4	12
Sample 1	249	7	24
Sample 2	217	4	28
Sample 3	170	3	22
Sample 4	131	3	13
Sample 5	101	2	14

^A The average of the laboratories' calculated averages.

8. Hazards

8.1 Consult current OSHA regulations, suppliers' Material 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

11.1 Prepare a synthetic mixture of high purity cyclohexane containing impurities at concentrations representative of those expected in the samples to be analyzed in accordance with Practice D4307. The weight of each hydrocarbon impurity must be measured to the nearest 0.1 mg. Because the availability of stock cyclohexane with a purity higher than 99.97 % is problematic, the method of standard additions may be required for impurities such as methylcyclohexane and methylcyclopentane, as well as for a number of the other impurities listed in Table 1 that are commonly present.

11.2 Inject the resulting solution from 11.1 into the gas chromatograph, collect and process the data. A typical chromatogram is illustrated in Fig. 1 based on the conditions listed in Table 2.

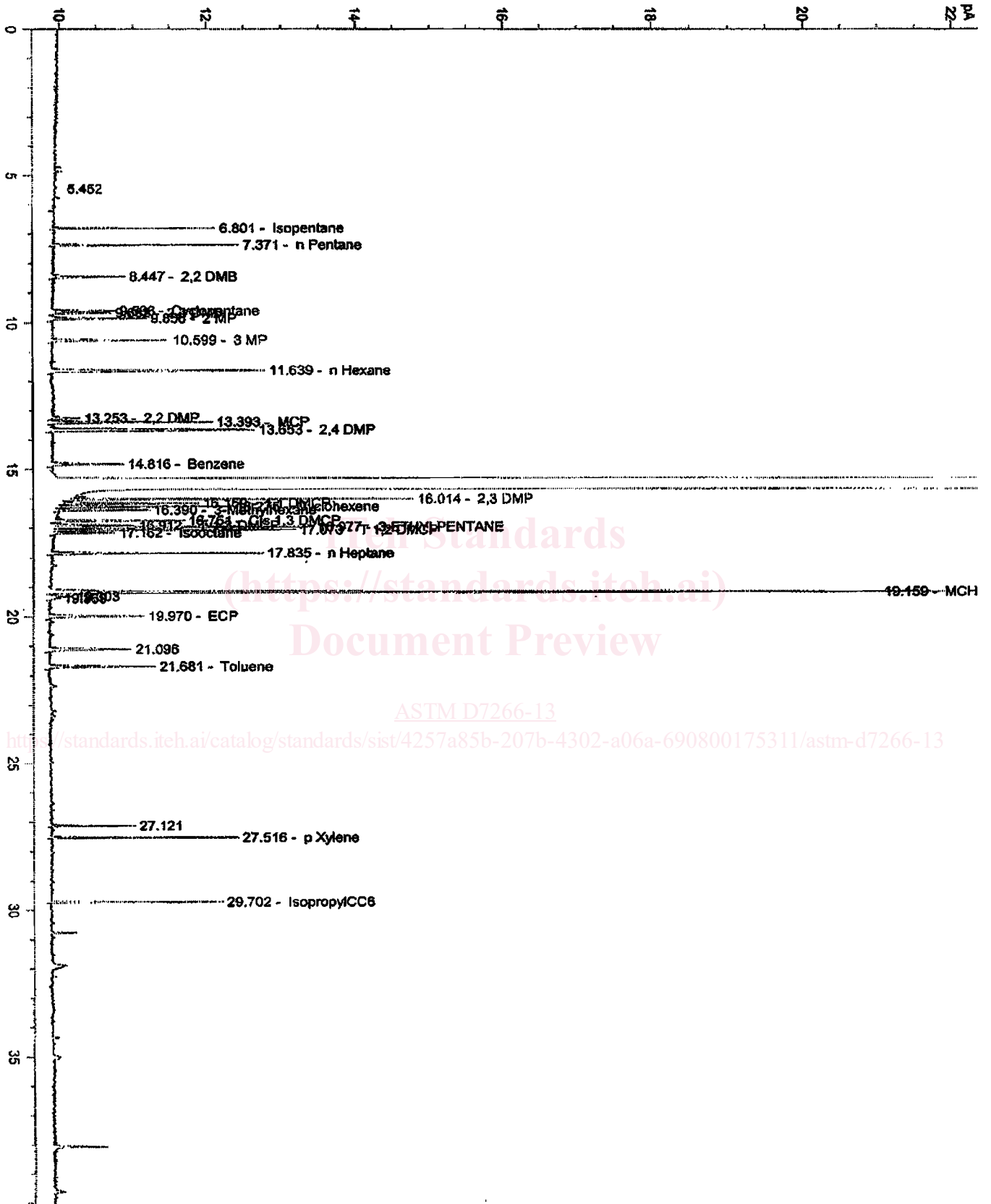


FIG. 1 Typical Chromatogram of Calibration Mixture Using Conditions in Table 2