Designation: D5713 – 14

Standard Test Method for Analysis of High Purity Benzene for Cyclohexane Feedstock by Capillary Gas Chromatography[1]

This standard is issued under the fixed designation D5713; 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 specific impurities in, and the purity of benzene for cyclohexane feedstock by gas chromatography.

1.2 This test method has been found applicable to benzene in the range from 99 to 100 % purity and to impurities at concentrations of 2 to 10 000 mg/kg.

1.3 In determining the conformance of the test results using this 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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see 7.2 and Section 8.

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

E260 Practice for Packed Column Gas Chromatography

E355 Practice for Gas Chromatography Terms and Relationships

E1510 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 *Definitions:*

3.1.1 See Terminology D4790 for definition of terms used in this test method.

4. Summary of Test Method

4.1 In this test method, the chromatogram peak area for each impurity is compared to the peak area of the internal standard (*n*-octane or other suitable known) added to the sample. From the response factor of toluene relative to that of the internal standard, and using a response factor of 1.00 for nonaromatic impurities and the amount of internal standard added, the concentrations of the impurities are calculated. The benzene content is obtained by subtracting the total amount of all impurities from 100.00.

5. Significance and Use

5.1 This test method is useful for benzene purity on the basis of impurities normally present in benzene and may be used for final product inspections and process control.

5.2 This test method will detect the following impurities: toluene, methylcyclopentane, *n*-hexane, 2-methylhexane, cyclohexane, cyclopentane, 2-methylpentane, 2,3-dimethylpentane, 3-methylhexane, *n*-heptane, methylcyclohexane, ethylcyclopentane, 2,4-dimethylhexane, trimethylpentane, and others where specific impurity standards

[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**

are available. Absolute purity cannot be accurately determined if unknown impurities are present.

6. Apparatus

6.1 *Gas Chromatograph*—Any gas chromatograph having a temperature programmable oven, flame ionization detector and a splitter injector suitable for use with a fused silica capillary column may be used, provided the system has sufficient sensitivity that will give a minimum peak height of 3 times the background noise for 2 mg/kg of an impurity.

6.2 *Column*—Fused silica capillary columns have been found to be satisfactory. An example is 50 m of 0.20-mm inside diameter fused silica capillary internally coated to a film thickness of 0.50 µm with polydimethylsiloxane (see Table 1 for suggested instrument parameters). Other columns may be used after it has been established that such a column is capable of separating all major impurities (for example, compounds listed in 5.2) and the internal standard from the benzene under operating conditions appropriate for the column. The column must give satisfactory resolution (distance from the valley between the peaks is not greater than 50 % of the peak heights of the impurity) of cyclohexane from benzene as well as other impurity peaks. A poorly resolved peak, such as cyclohexane, will often require a tangent skim from the neighboring peak.

6.3 *Electronic Integration,* with tangent skim capabilities is recommended.

6.4 *Vial.*

6.5 *Microsyringes,* assorted volumes.

6.6 *Injector,* the specimen must be precisely and repeatedly injected into the gas chromatograph. An automatic sample injection device is highly recommended. Manual injection can be employed if the precision stated in Table 2 can be reliably and consistently satisfied.

TABLE 1 Recommended Operating Conditions

Carrier gas	hydrogen
Linear velocity at 40°C, cm/s	40
Detector	flame ionization
Detector temperature, °C	250°C
Injection port temperature, °C	250°C
Split ratio	40:1
Split flow, mL/min	60
Column	50 m by 0.20 mm ID by 0.5 µm bonded polydimethylsiloxane capillary column
Initial column temperature, °C	40
Initial time, min	17
Programming rate	10°C/min
Final temperature, °C	250°C
Final time, min	10
Sample size, µL	1.2

TABLE 2 Summary of Precision Data

Component	Average Concentration mg/kg	Intermediate Precision	Reproducibility
Benzene (weight %)	99.96	0.006	0.022
	99.97	0.007	0.020
	99.96	0.008	0.025
Methylcyclopentane	104	8.3	27.9
	43	12.2	19.4
	54	2.5	15.1
Toluene	64	5.1	22.0
	63	3.0	16.6
	28	1.8	9.1
Methylcyclohexane	132	7.4	34.8
	43	1.4	5.4
	79	3.2	17.0
Methylcyclohexane + Toluene	196	7.9	54.9
	106	12.9	33.6
	106	4.4	20.4
n-Hexane	4	2.2	3.7
	3	1.5	2.2
	2	1.8	2.5
n-Heptane	6	2.7	11.1
	16	1.5	5.6
	15	4.0	23.4
Ethylcyclopentane	7	1.8	3.7
	6	1.9	11.0
	11	1.5	6.1
Total Other Impurities	99	22.5	163.0
	107	44.6	190.6
	185	55.5	233.0

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 *Carrier Gas*—a carrier gas (99.999 % minimum purity) appropriate to the type of detector used should be employed. (**Warning**—If hydrogen is used as the carrier gas, take special safety precautions to ensure that the system is free of leaks and that the effluent is properly vented or burned.)

7.3 *Hydrogen,* for the flame ionization detector (FID) minimum purity of 99.999 % and <0.5 ppm total hydrocarbons is preferred.

7.4 *Air,* for the flame ionization detector, <0.1 ppm total hydrocarbons is preferred.

7.5 *n-octane,* 99.0 % minimum purity, or other internal standard (99.0 % minimum purity), such as *iso*-octane, previously analyzed to be free of compounds coeluting with impurities in the sample.

[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*, BHD, Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.