Designation: D7057 – 13

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

This standard is issued under the fixed designation D7057; 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 isopropylbenzene (cumene) 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 nonaromatic hydrocarbons, benzene, toluene, ethylbenzene, phenol, cymenes, *t*-butylbenzene, *n*-butylbenzene, *n*-propylbenzene, *alpha*-methylstyrene, *sec*-butylbenzene, and diisopropylbenzenes, which are impurities that can be found in isopropylbenzene. The latter impurities can be analyzed over a range of 1 mg/kg to 500 mg/kg by this method (see Table 1). The limit of detection for these impurities is summarized in Table 1 as well.

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 Section 8.

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
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 paragraphs 1910.1000 and 1910.1200[3]

3. Terminology

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

4. Summary of Test Method

4.1 Cumene (isopropylbenzene) is analyzed by 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 Cumene content) is calculated by subtracting the sum of the impurities from 100.00, excluding Acetophenone (AP) and 2-phenyl-2-propanol (DMPC or DMBA) which are artifacts of cumene hydroperoxide (CHP) thermal decomposition in the inlet of the GC. Results are reported in weight percent.

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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 D16.07 on Styrene, Ethylbenzene and C9 and C10 Aromatic Hydrocarbons.

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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**

TABLE 1 Summary of Precision Data (mg/kg)

Compound	Repeatability	Reproducibility	Range Studied
Benzene	1.004+0.006*Benzene	0.924+0.158*Benzene	1.0–295
Toluene	0.644+0.009*Toluene	1.505+0.069*Toluene	0.2–260
Ethylbenzene (EB)	1.249+0.022*EB	2.534+0.230*EB	5–370
n-Propylbenzene (NPB)	-0.369+0.015*NPB	-6.793+0.214*NPB	180–465
Phenol (PHE)	1.926+.0095*PHE	3.718+0.207*PHE	1–225
alpha-methylstyrene(AMS)	1.368+0.057*AMS	21.181+0.976*AMS	6–305
tert-Butylbenzene(TBB)	1.666+0.011*TBB	4.126+0.086*TBB	2–250
sec-Butylbenzene(SBB)	0.635+0.010*SBB	0.891+0.090*SBB	2–245
m-Cymene	0.618+0.011**m*-Cymene	1.811+0.181**m*-Cymene	5–240
o-Cymene	0.498+0.012**o*-Cymene	1.951+0.156**o*-Cymene	1–160
p-Cymene	0.485+0.019**p*-Cymene	1.618+0.097**p*-Cymene	2–270
n-Butylbenzene(NBB)	0.445+0.030*NBB	2.600+0.045*NBB	0.3–265
1,3-diisopropylbenzene(13DIPB)	0.727+0.010*13DIPB	1.570+0.720*13DIPB	1–210
1,4-diisopropylbenzene(14DIPB)	0.435+0.013*14DIPB	1.112+0.040*14DIPB	1–285
NonAromatics(NA)	0.441+0.242*NA	1.663+0.306*NA	5–95

5. Significance and Use

5.1 This test method is suitable for setting specifications on the materials referenced in 1.2 and for use as an internal quality control tool where isopropylbenzene is produced or is used in a manufacturing process. It may also be used in development or research work involving isopropylbenzene.

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

5.3 Cumene hydroperoxide, if present, will yield thermal decomposition products, primarily AP and DMPC as stated in 4.1, that will elute in the chromatogram thereby giving incorrect results unless they are excluded as indicated in 4.1. It is also possible to get erroneous results for trace *alpha*-methylstyrene present in isopropylbenzene if the inlet has become unduly contaminated with non-volatile foreign substances.

5.4 The nonaromatic hydrocarbons commonly present from the isopropylbenzene manufacturing process, particularly a non-zeolitic one, will interfere with the determination of xylenes (if present).

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 isopropylbenzene. The column described in Table 2 has been used successfully and shall be used as a referee in cases of dispute. Unless the analyst can be sure of peak identity, for example by gas chromatography-mass spectrometry (GC-MS), the use of the column in Table 2 is strongly recommended.

6.3 *Recorder*—Electronic integration is required.

TABLE 2 Recommended Operating Conditions

Detector	flame ionization
Injection Port	capillary splitter
Column A:	
Tubing	fused silica
Stationary Phase	crosslinked polydimethylsilicone (HP-1, DB-1 or equiv)
Film Thickness, µm	1.0
Length, m	60
Diameter, mm	0.32
Temperatures:	
Injector, °C	250
Detector, °C	300
Oven, °C	85 hold for 13 min Ramp 1 = 6°C/min to 125°C, hold for 2 min Ramp 2 = 30°C/min to 250°C, hold for 7.5 min
Carrier Gas	Helium
Flow Rate, mls/min	3
Split Ratio	60:1
Sample Size, µL	1.0

6.4 *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 1, Summary of Precision Data, 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.

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[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.

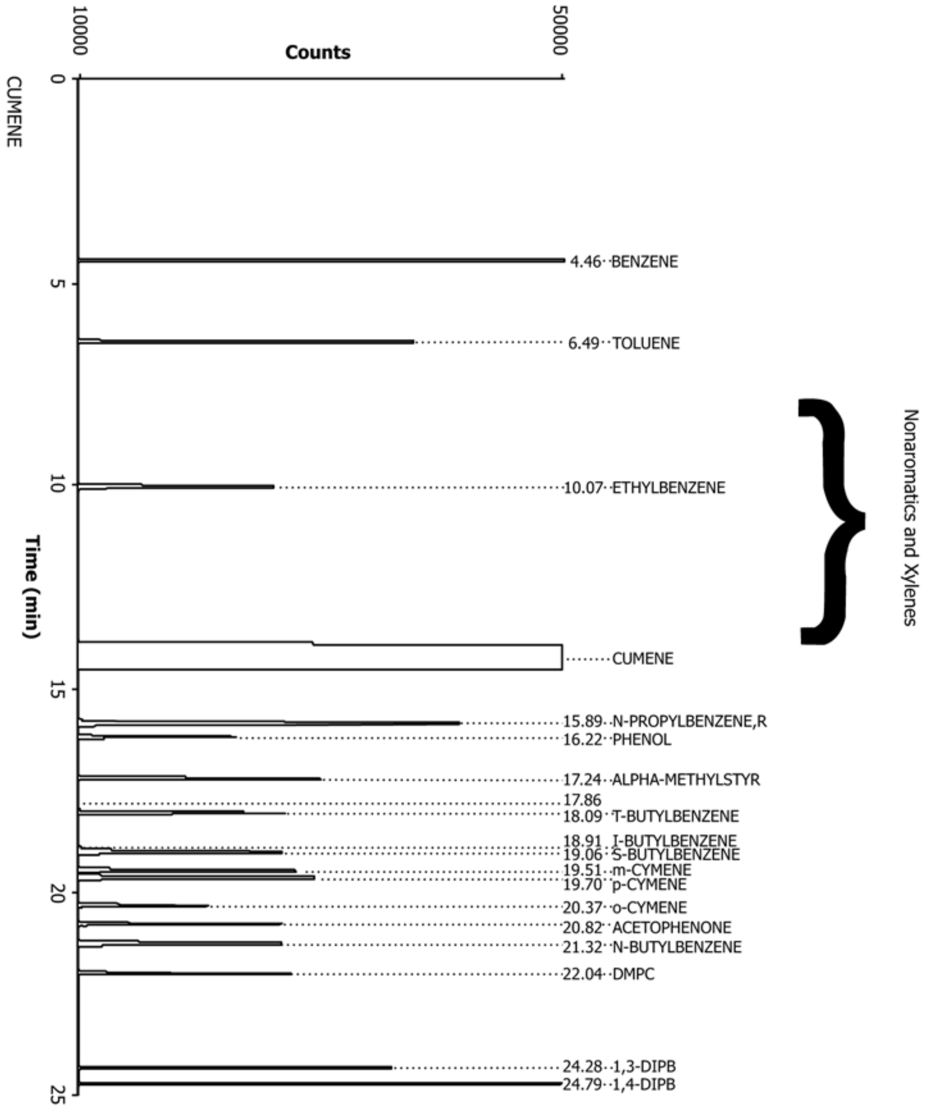

FIG. 1 Typical Chromatogram using Conditions in Table 2

7.2 *Carrier Gas, Makeup, and Detector Gases*—Helium, hydrogen, nitrogen, or other carrier, makeup and detector gases 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.

7.3 *Compressed Air*—Purify air to remove water and hydrocarbons. Air for a FID should contain less than 0.1 ppm THC.

7.4 *Pure Compounds for Calibration*—The purity of all reagents should be 99.9 % or greater. If the purity is less than 99 % the concentration and identification of impurities must be known so that the composition of the standard can be adjusted for the presence of the impurities.

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 Practice E355 for additional information on gas chromatography practices and terminology.

11. Calibration

11.1 Prepare a synthetic mixture of high purity cumene 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 cumene with a purity higher than 99.97 % is problematic, the method of standard additions may be required for impurities such as *tert*-butylbenzene and *n*-propylbenzene, as well as for a number of the other impurities listed in 1.2 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.

11.3 Determine the response factor for each impurity in the calibration mixture as follows:

$$Rfi = Ci/Ai \tag{1}$$

where:

Rfi = response factor for impurity i,
Ci = concentration of impurity i in the calibration mixture, and
Ai = peak area of impurity i.

11.4 Initially analyze the calibration solution a minimum of three times and calculate an average Rfi. Subsequent calibrations may be a single analysis as long as the response factors for all components of interest are within ±5 % of the initial validation response factors. A "rolling" average as defined by most modern chromatographic software may also be used. The response factor for *tert*-butylbenzene is used for unknowns and non-aromatics quantification.

12. Procedure

12.1 Inject into the gas chromatograph an appropriate amount of sample sufficient to satisfy the sensitivity conditions as detailed in 6.1 and start the analysis.

12.2 Obtain a chromatogram and peak integration report. Fig. 1 illustrates a typical analysis of cumene using the column and conditions outlined in Table 2.

13. Calculations

13.1 Calculate the concentration of each impurity as follows:

$$Ci = (Ai)\,(Rfi) \tag{2}$$

where:

Ci = concentration of component i in mg/kg,
Ai = peak area of component i, and
Rfi = response factor for component i,

13.2 Calculate the total concentration of all impurities in wt % (excluding AP and DMBA as per 5.3) as follows:

$$C_t = \sum C_i/10000 \tag{3}$$

where:

C_t = total concentration of all impurities.

13.3 Calculate the purity of isopropylbenzene as follows:

$$\text{Cumene, weight percent} = 100.00 - C_t \tag{4}$$

14. Report

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

14.2 Report the purity of isopropylbenzene to the nearest 0.01 wt %.

15. Precision and Bias[5]

15.1 The criteria in Table 1 should be used to judge the acceptability at the 95 % probability level of the results obtained by this test method. The criteria were derived from an Interlaboratory study of five samples analyzed in triplicate by five laboratories. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D16-1033.

15.2 *Repeatability*—Results in the same laboratory should not be considered suspect unless they differ by more than the amounts calculated from the appropriate equations in Table 1. Results differing by less than "*r*" have a 95 % probability of being correct.

15.3 *Reproducibility*—Results submitted by two laboratories should not be considered suspect unless they differ by more than the amounts calculated from the appropriate equations in Table 1. Results differing by less than "*R*" have a 95 % probability of being correct.

15.4 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method for measuring isopropylbenzene purity, bias has not been determined.

16. Quality Guidelines

16.1 Laboratories shall have a quality control system in place.

16.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

16.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

16.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

16.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical quality control practices.

17. Keywords

17.1 *alpha*-methylstyrene; analysis by gas chromatography; benzene; butylbenzene; cumene; cymene; diisopropylbenzene; ethylbenzene; isopropylbenzene; methyl cumene; nonaromatic hydrocarbons; propylbenzene; toluene

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1033. Contact ASTM Customer Service at service@astm.org.

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D7057–09) that may impact the use of this standard. (Approved June 1, 2013.)

(1) All sections updated to current D16 Editorial Guidelines.

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