



Designation: **D7057–09 D7057 – 13**

## Standard Test Method for Analysis of Isopropylbenzene (Cumene) by Gas Chromatography (External Standard)<sup>1</sup>

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 ( $\epsilon$ ) 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:<sup>2</sup>

**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, 29CFR paragraphs 1910.1000 and 1910.1200**<sup>3</sup>

### 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

<sup>1</sup> 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 C<sub>9</sub> and C<sub>10</sub> Aromatic Hydrocarbons.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> 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
<i>n</i> -Propylbenzene (NPB)	-0.369+0.015*NPB	-6.793+0.214*NPB	180–465
Phenol (PHE)	1.926+0.0095*PHE	3.718+0.207*PHE	1–225
<i>alpha</i> -methylstyrene(AMS)	1.368+0.057*AMS	21.181+0.976*AMS	6–305
<i>tert</i> -Butylbenzene(TBB)	1.666+0.011*TBB	4.126+0.086*TBB	2–250
<i>sec</i> -Butylbenzene(SBB)	0.635+0.010*SBB	0.891+0.090*SBB	2–245
<i>m</i> -Cymene	0.618+0.011* <i>m</i> -Cymene	1.811+0.181* <i>m</i> -Cymene	5–240
<i>o</i> -Cymene	0.498+0.012* <i>o</i> -Cymene	1.951+0.156* <i>o</i> -Cymene	1–160
<i>p</i> -Cymene	0.485+0.019* <i>p</i> -Cymene	1.618+0.097* <i>p</i> -Cymene	2–270
<i>n</i> -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

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.

## 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. 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.

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.<sup>4</sup> 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.

<sup>4</sup> *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 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, $\mu\text{m}$	1.0
Length, m	60
Diameter, mm	0.32
Temperatures:	
Injector, $^{\circ}\text{C}$	250
Detector, $^{\circ}\text{C}$	300
Oven, $^{\circ}\text{C}$	85 hold for 13 min Ramp 1 = $6^{\circ}\text{C}/\text{min}$ to $125^{\circ}\text{C}$ , hold for 2 min Ramp 2 = $30^{\circ}\text{C}/\text{min}$ to $250^{\circ}\text{C}$ , hold for 7.5 min
Carrier Gas	Helium
Flow Rate, mls/min	3
Split Ratio	60:1
Sample Size, $\mu\text{L}$	1.0

7.2 Carrier Gas, Makeup, and Detector Gases—Helium, Hydrogen, Nitrogen, hydrogen, nitrogen, or other as Carrier. Carrier, makeup, 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. Purify air to remove hydrocarbons and water, and the air used should be zero grade or better.

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 Practices Practice E1510 and 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:

$$Rf_i = C_i/A_i \quad (1)$$

where:

$Rf_i$  = response factor for impurity  $i$ ,

$C_i$  = concentration of impurity  $i$  in the calibration mixture, and

$A_i$  = peak area of impurity  $i$ .