ASTM INTERNATIONAL

Designation: D7057 – 18

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 quantitation averages 3.8 mg/kg while the limit of detection for these impurities averages 1.1 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard 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

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

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*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

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

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

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*—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. Recorders are not considered adequate for meeting the precision requirements of this standard.

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