Designation: D3760 – 12

StandardTest Method for Analysis of Isopropylbenzene (Cumene) by Gas Chromatography[1]

This standard is issued under the fixed designation D3760; 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.

1.2 This test method has been found applicable to the measurement of impurities such as nonaromatic hydrocarbons, benzene, ethylbenzene, *t*-butylbenzene, *n*-propylbenzene, alpha-methylstyrene, sec-butylbenzene, and diisopropylbenzene, which are common to the manufacturing process of isopropylbenzene. Limit of detection for these impurities is 10 mg/kg (see 5.1). This method has been found applicable for concentrations of various components up to 571 ppm.

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 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3437 Practice for Sampling and Handling Liquid Cyclic Products

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

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. Summary of Test Method

3.1 A known amount of internal standard is added to a sample of isopropylbenzene. The prepared sample is mixed and analyzed by a gas chromatograph equipped with a flame ionization detector (FID). The peak area of each impurity and the internal standard is measured and the amount of each impurity is calculated from the ratio of the peak area of the internal standard versus the peak area of the impurity. Purity by GC (the isopropylbenzene content) is calculated by subtracting the sum of the impurities found from 100.00. Results are reported in weight percent.

4. Significance and Use

4.1 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.

4.2 This test method is useful in determining the purity of isopropylbenzene with normal impurities present including diisopropylbenzenes. If extremely high boiling or unusual

[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 Recommended Operating Conditions

	Column A	Column B
Detector	Flame Ionization	Flame Ionization
Column:		
Tubing	fused silica	fused silica
Stationary phase	polyethylene glycol	methyl silicone
Solid support	crosslinked	crosslinked
Film thickness	0.25 µ	0.5 µ
Length, m	50	50
Diameter, mm	0.32 mm ID	0.32 mm ID
Temperatures:		
Injector, °C	275	275
Detector, °C	300	300
Oven:		
Initial, °C	60	35
Time 1, min	10	10
Final, °C	175	275
Rate, °C/min	10	5
Time 2, min	10	0
Carrier gas	hydrogen	helium
Flow rate, mL/min	1.0	1.0
Split ratio	100:1	100:1
Sample size, µL	1.0	1.0

impurities are present in the isopropylbenzene, this test method would not necessarily detect them and the purity calculation would be erroneous.

4.3 Cumene hydroperoxide, if present, will yield decomposition products that will elute in the chromatogram thereby giving incorrect results.

4.4 The nonaromatic hydrocarbons commonly present from the isopropylbenzene manufacturing process will interfere with the determination of benzene when Column A in Table 1 is used.

4.5 The internal standard must be sufficiently resolved from any impurity and the isopropylbenzene peak.

5. Apparatus

5.1 *Gas Chromatograph*—Any instrument having a flame ionization detector that can be operated at the conditions given in Table 1. The system should have sufficient sensitivity to obtain a minimum peak height response for 10 mg/kg *n*-butylbenzene of twice the height of the signal background noise.

5.2 *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 and from the internal standard. The columns and conditions described in Table 1 have been used successfully and shall be used as referee in cases of dispute.

5.3 *Recorder*—Electronic integration is recommended.

6. Reagents and Materials

6.1 *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.

6.1.1 *Internal Standard*—Normal Butylbenzene (*n*BB) is the recommended internal standard of choice. Other compounds may be found acceptable provided they meet the criteria as defined in 4.5 and 6.1.

6.2 *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.

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

6.4 *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.

7. Hazards

7.1 Consult current OSHA regulations and suppliers' Material Safety Data Sheets on handling materials listed in this test method.

8. Sampling and Handling

8.1 Sample the material in accordance with Practice D3437.

9. Preparation of Apparatus

9.1 Follow manufacturer's instructions for mounting and conditioning the column into the chromatograph and adjusting the instrument to the conditions described in Table 1 allowing sufficient time for the equipment to reach equilibrium. See Practice E1510 for more information on column installation. See Practice E355 for additional information on gas chromatography practices and terminology.

10. Procedure

10.1 Into a 100-mL volumetric flask, add 100 µL of *n*BB to 99.90 mL of cumene. Mix well. Assuming a density of 0.856 for *n*BB and 0.857 for cumene, the resulting *n*BB concentration will be 0.1000 weight %.

10.2 Inject into the gas chromatography an appropriate amount of sample as previously determined according to 6.1 and start the analysis.

10.3 Obtain a chromatograph and peak integration report. Fig. 1 and Fig. 2 illustrate a typical analysis of cumene for Columns A and B, respectively.

[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.

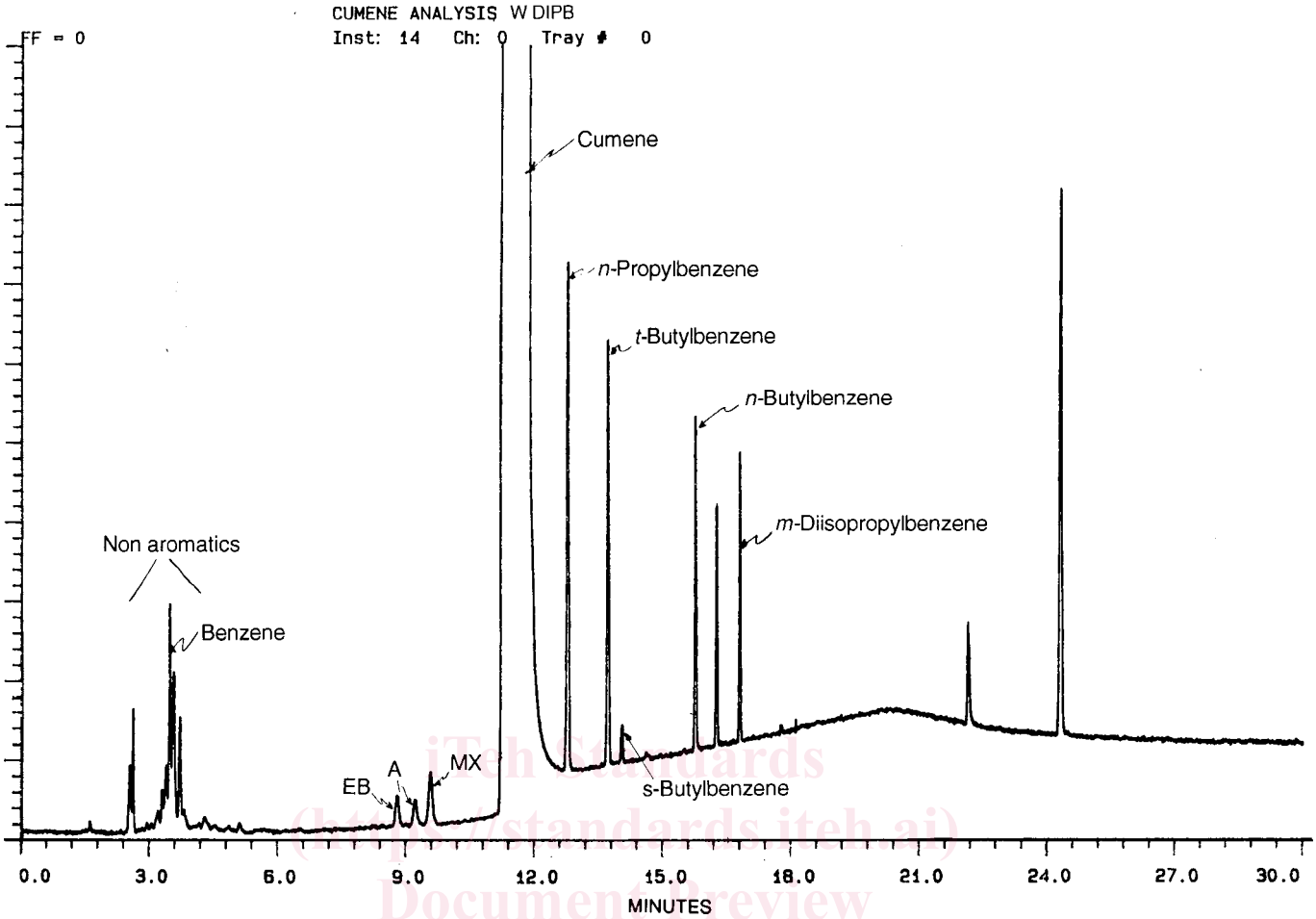

FIG. 1 Typical Chromatogram using Conditions for Column A

11. Calculations

11.1 Determine the area defined by each peak in the chromatogram.

11.2 Calculate the percent concentration of the total nonaromatics and each impurity as follows:

$$C_i = \frac{(A_i)(C_2)}{(A_2)} \quad (1)$$

where:

C_i = concentration of component i, weight %,
A_i = peak area of component i,
A_2 = peak area of *n*BB,
C_2 = concentration of *n*BB, weight %.

11.3 Calculate the total concentration of all impurities as follows:

$$C_t = \sum C_i \quad (2)$$

where:

C_t = total concentration of all impurities.

11.4 Calculate the purity of isopropylbenzene as follows:

$$\text{isopropylbenzene, weight \%} = 100.000 - C_t \quad (3)$$

12. Report

12.1 Report the individual impurities to the nearest 0.0001 %.

12.2 Report the purity of isopropylbenzene to the nearest 0.001 weight%.

13. Precision and Bias[5]

13.1 *Precision*—The following criteria should be used to judge the acceptability of results obtained by this test method (95 % confidence level). The precision criteria were derived from an ILS that was conducted using the conditions listed under Column B in Table 1 which included nine laboratories analyzing four samples in triplicate. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D16-1032.

13.1.1 *Intermediate Precision (formerly called Repeatability)*—Results should not be suspect unless they differ by more than the amount listed in Table 2 and Table 3. Results differing by less than "*r*" have a 95 % probability of being correct.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1032.