



Designation: D 3760 – 98

Standard Test Method for Analysis of Isopropylbenzene (Cumene) by Gas Chromatography¹

This standard is issued under the fixed designation D 3760; 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).

1.3 The following applies to all specified limits in this standard: for purposes of determining conformance with this standard, an observed value or a calculated value shall be rounded off “to the nearest unit” in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

1.4 *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:

D 3437 Practice for Sampling and Handling Liquid Cyclic Products²

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³

E 260 Practice for Packed Gas Chromatography³

E 355 Practice for Gas Chromatography Terms and Relationships³

2.2 Other Document:

OSHA Regulations, 29CFR, paragraphs 1910.1000 and 1910.1200⁴

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 D16.0H on Styrene, Ethylbenzene, and C₉ and C₁₀ Aromatic Hydrocarbons.

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² *Annual Book of ASTM Standards*, Vol 06.04.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

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 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

TABLE 1 Instrumental Parameters

	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, μ L	1.0	1.0

from the internal standard. The columns described in Table 1 have been used successfully.

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.⁵ 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*—Chromatographic grade helium or hydrogen is recommended.

6.3 *Compressed Air*—Chromatographic grade.

6.4 *Hydrogen*—High purity.

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 D 3437.

9. Preparation of Apparatus

9.1 Follow manufacturer's instructions for mounting and

⁵ *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.

† Editorially corrected.

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 Practices E 260 and E 355 for additional information on gas chromatography practices and terminology.

10. Procedure

10.1 Into a 100-mL volumetric flask, add 100 μ L 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.

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

13.1 *Precision*—The criteria in Table 2 should be used to judge the acceptability of results obtained by this test method. The criteria were derived from round robin testing of four samples. Table 3 illustrates equations for repeatability and reproducibility.

13.2 *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.

14. Keywords

14.1 alpha methylstyrene; benzene; butylbenzene; cumene; ethylbenzene; isopropylbenzene; nonaromatic hydrocarbons;

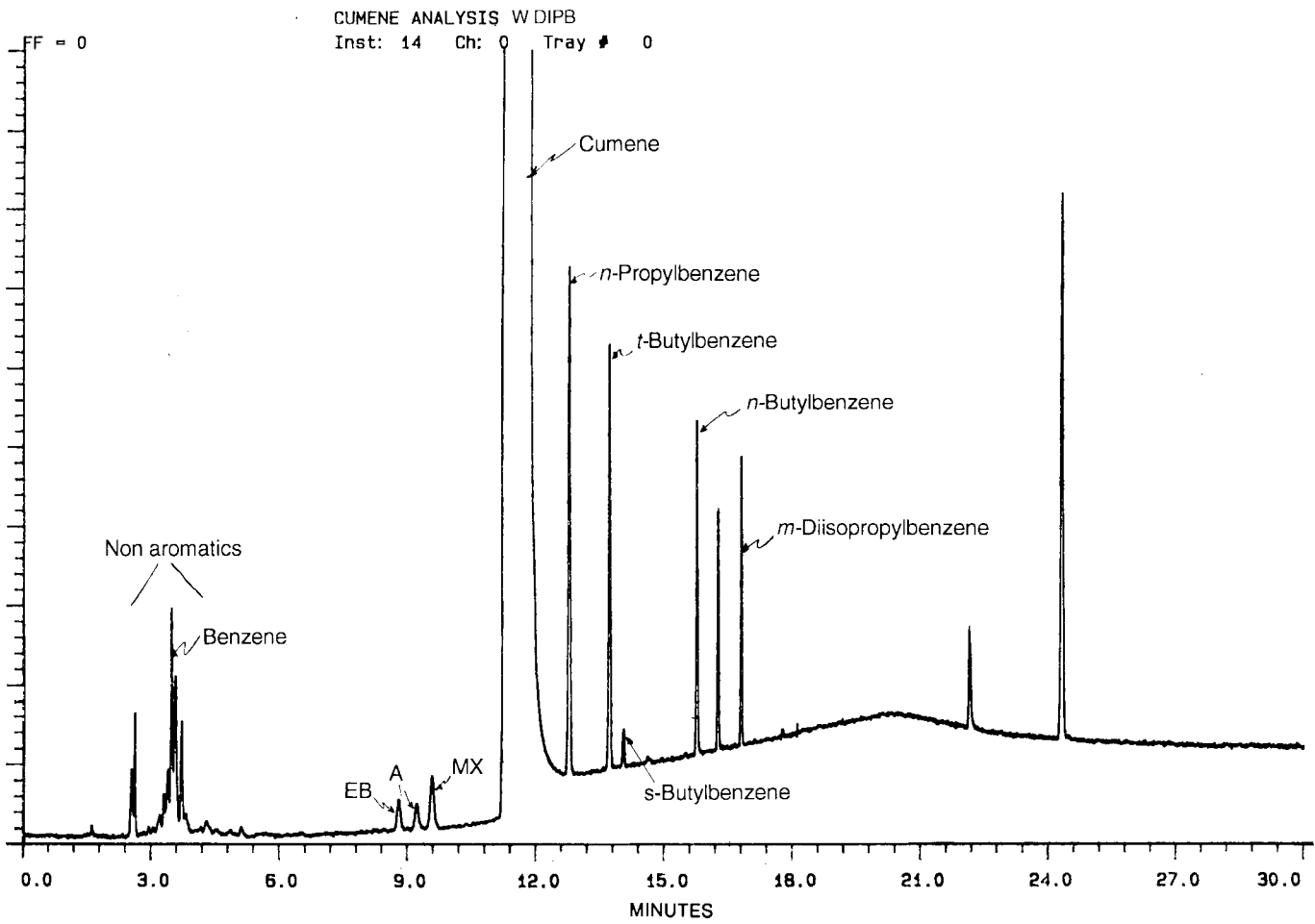


FIG. 1 Typical Chromatogram using Conditions for Column A

propylbenzene; analysis by gas chromatography

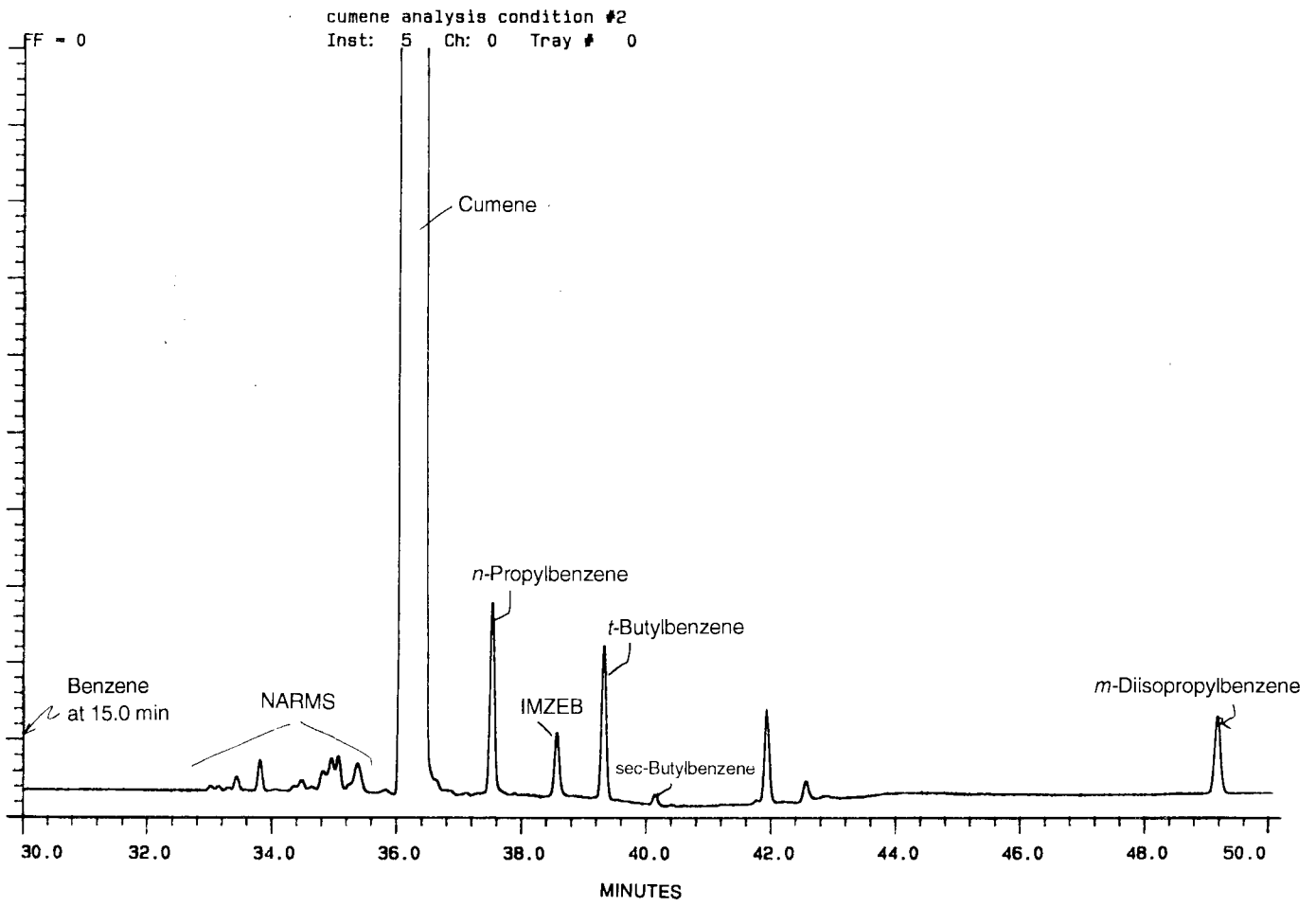



FIG. 2 Typical Chromatogram using Conditions for Column B

TABLE 2 Typical Values for Repeatability and Reproducibility for Selected Cumene Components

Component	Concentration, wt %	Repeatability	Reproducibility
<i>a</i> -Methylstyrene	0.0065	0.00048	0.00072
	0.0083	0.00048	0.00074
	0.0104	0.00131	0.00151
Benzene	0.0024	0.00023	0.00088
Ethylbenzene	0.0042	0.00046	0.00350
	0.0080	0.00050	0.00298
	0.0099	0.00111	0.00519
Nonaromatics	0.0163	0.00231	0.00482
	0.0571	0.00635	0.00635
<i>n</i> -Propylbenzene	0.0192	0.00170	0.00169
	0.0218	0.00113	0.00145
	0.0246	0.00006	0.00088
<i>s</i> -Butylbenzene	0.0024	0.00014	0.00082
	0.0037	0.00030	0.00063
	0.0059	0.00034	0.00071
<i>t</i> -Butylbenzene	0.0067	0.00041	0.00110
	0.0106	0.00031	0.00106
	0.0283	0.00102	0.00353
Cumene	99.8443	0.00237	0.09720
	99.8657	0.01050	0.06220
	99.9221	0.01300	0.04290
	99.9386	0.01060	0.02750

TABLE 3 Repeatability and Reproducibility

Cumene	Repeatability = $0.07801(x) - 7.7843$ Reproducibility = $-0.63789(x) + 63.7785$
<i>t</i> -Butylbenzene	Repeatability = $0.031894(x) + 0.00009$ Reproducibility = $0.120596(x) + 0.0000648$
<i>s</i> -Butylbenzene	Repeatability = $0.054069(x) + 0.0000437$ Reproducibility = $-9.60703(x) + 0.000817$
<i>n</i> -Propylbenzene	Repeatability = $-0.30407(x) + 0.007613$ Reproducibility = $-0.14927(x) + 0.004605$
<i>a</i> -Methylstyrene	Repeatability = $0.21785(x) - 0.0010752$ Reproducibility = $0.20833(x) - 0.000761$
Benzene	Repeatability = $0.095(x)$ Reproducibility = $0.367175(x)$
Nonaromatics	Repeatability = $0.098932(x) - 0.000697$ Reproducibility = $0.03733(x) + 0.004214$
Ethylbenzene	Repeatability = $0.099441(x) - 0.000045$ Reproducibility = $0.208332(x) + 0.0021557$

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