Designation: D 5060 – 95 (Reapproved 2000)

Standard Test Method for Determining Impurities in High-Purity Ethylbenzene by Gas Chromatography[1]

This standard is issued under the fixed designation D 5060; 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 describes the analysis of normally occurring impurities in, and the purity of, ethylbenzene by gas chromatography. Impurities determined include nonaromatic hydrocarbons, benzene, toluene, xylenes, cumene, and diethylbenzene isomers.

1.2 This test method is applicable for impurities at concentrations from 0.001 to 1.000 % and for ethylbenzene purities of 99 % or higher. At this level, *p*-xylene may not be detected.

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 a specific hazard statement, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 3437 Practice for Sampling and Handling Liquid Cyclic Products[2]

D 4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards[3]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[4]

E 355 Practice for Gas Chromatography Terms and Relationships[4]

E 1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs[4]

2.2 *Other Documents:*

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200[5]

3. Summary of Test Method

3.1 A known amount of internal standard is added to the sample. A gas chromatograph equipped with a flame ionization detector and a polar fused silica capillary column is used for the analysis. The impurities are measured relative to the internal standard. Ethylbenzene purity is calculated by subtracting the impurities found from 100.00 %.

4. Significance and Use

4.1 The test is suitable for setting specifications on ethylbenzene and for use as an internal quality control tool where ethylbenzene is used in manufacturing processes. It may be used in development or research work involving ethylbenzene.

4.2 Purity is commonly reported by subtracting the determined expected impurities from 100 %. Absolute purity cannot be determined if unknown impurities are present.

5. Apparatus

5.1 *Gas Chromatograph*—Any gas chromatograph having a flame ionization detector and a splitter injector suitable for use with a fused-silica capillary column may be used, provided the system has sufficient sensitivity to obtain a minimum peak height response of 0.1 mV for 0.010 % internal standard when operated at the stated conditions. Background noise at these conditions is not to exceed 3 µV.

5.2 *Chromatographic Column*, fused silica capillary, 60 m long, 0.32-mm inside diameter, internally coated to a 0.5-µm thickness with a bonded (crosslinked) polyethylene glycol. Other columns may be used after it has been established that such column is capable of separating all major impurities and the internal standard from the ethylbenzene under operating conditions appropriate for the column.

5.3 *Recorder*, 1-mV, 1 s or less full scale response or electronic integration with tangent capabilities (recommended).

5.4 *Microsyringe*, 10-µL.

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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 C_9 and C_{10} Aromatic Hydrocarbons.

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

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 2O402.

5.5 *Microsyringe*, 50-µL.

5.6 *Volumetric Flask*, 50-mL.

6. Reagents and Materials

6.1 *Carrier Gas*, hydrogen or helium, chromatographic grade.

6.2 *Compressed Air*, oil-free.

6.3 *Hydrogen*, chromatographic grade.

6.4 *Nitrogen*, chromatographic grade.

6.5 *Pure Compounds for Calibration*—*n*-Nonane, benzene, toluene, ethylbenzene, and *o*-xylene. The purity of the ethylbenzene should be 99.8 % or better. The ethylbenzene must be analyzed and corrections made in the composition of the calibration blend as required. The purity of all other compounds should be 99 % or greater. If the purity is less than 99 %, the concentration and identification of the impurities must be known so that the composition of the calibration standard can be adjusted for the presence of the impurities.

6.6 *n-Undecane*, for use as internal standard, 99 % or greater purity.

7. Hazards

7.1 Consult current OSHA regulations, supplier's Material Safety Data Sheets, and local regulations for all materials used in this test method.

8. Sampling

8.1 Guidelines for taking samples from bulk are given in Practice D 3437.

9. Calibration

9.1 Prepare a calibration blend of each compound listed in 6.5 and *n*-undecane at the 0.2 weight % level in ethylbenzene as described in Practice D 4307. *n*-Nonane represents the nonaromatic hydrocarbons. A series of calibration blends that span the concentration range should be prepared, one at the expected level of impurities, another at one half the expected level, and a third series at twice the expected level.

9.2 Analyze the ethylbenzene used in preparing the calibration blend as described in 10.3.

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9.4 Calculate response factors as follows:

$$R_i = \frac{C_i}{(C_s)\left(\frac{A_i}{A_{s,i}} - \frac{A_b}{A_{s,b}}\right)} \qquad (1)$$

where:

R_i = response factor for impurity relative to internal standard,

A_i = area of impurity peak in calibration blend,

A_b = area of impurity in ethylbenzene in calibration blend,

C_s = concentration of internal standard, weight %,

$A_{s,i}$ = area of internal standard peak in calibration blend,

$A_{s,b}$ = area of internal standard peak in stock ethylbenzene, and

C_i = concentration of impurity, weight %.

9.5 Calculate response factor to the nearest 0.001.

10. Procedure

10.1 Install the chromatographic column and establish stable instrument operation at the operating conditions shown in Table 1. Refer to instructions provided by the manufacturer of the gas chromatograph and Practices E 355 and E 1510.

10.2 Fill a 50-mL volumetric flask to the mark with test specimen. With a microsyringe, add 30 µL of the standard. Mix well. Using a density of 0.740 for *n*-undecane and 0.867 for ethylbenzene, this solution will contain 0.0512 weight % internal standard.

10.3 Inject 0.6 µL of solution into the gas chromatograph and obtain the chromatogram. A typical chromatogram is shown in Fig. 1.

11. Calculation

11.1 Measure the areas of all peaks, including the internal standard, except for the ethylbenzene peak.

11.2 Sum all the peaks eluting before ethylbenzene except for benzene, toluene, and the internal standard. Identify this sum as nonaromatic hydrocarbons.

11.3 Calculate the weight percent of the individual impurities, C_i, to the nearest 0.001 %, as follows:

$$C_i = \frac{0.0512\, A_i R_i}{A_s} \qquad (2)$$

where:

A_i = area of impurity,

R_i = response factor for impurity, and

A_s = area of internal standard.

11.4 Use the response factor determined for *o*-xylene for all the peaks eluting after ethylbenzene, and the response factor determined for *n*-nonane for all the nonaromatic hydrocarbon peaks.

11.5 Calculate the purity of the ethylbenzene by subtracting the sum of the impurities from 100.00.

12. Report

12.1 Report the following information:

12.1.1 The concentration of each impurity to the nearest 0.001 weight %, and

12.1.2 The purity of ethylbenzene to the nearest 0.01 weight %.

13. Precision and Bias

13.1 The following criteria should be used to judge the acceptability of the 95 % probability level of the results

TABLE 1 Typical Instrument Parameters

Carrier gas	helium
Carrier gas flow rate at 110°C, mL/min	1.2
Detector	flame ionization
Detector temperature, °C	240
Injection port temperature, °C	230
Hydrogen flow rate, mL/min	30
Airflow rate, mL/min	275
Make-up gas	nitrogen
Make-up gas flow rate, mL/min	23
Split flow, mL/min	150
Column temperature, °C	110
Chart speed, cm/min	1
Sample size,µ L	0.6

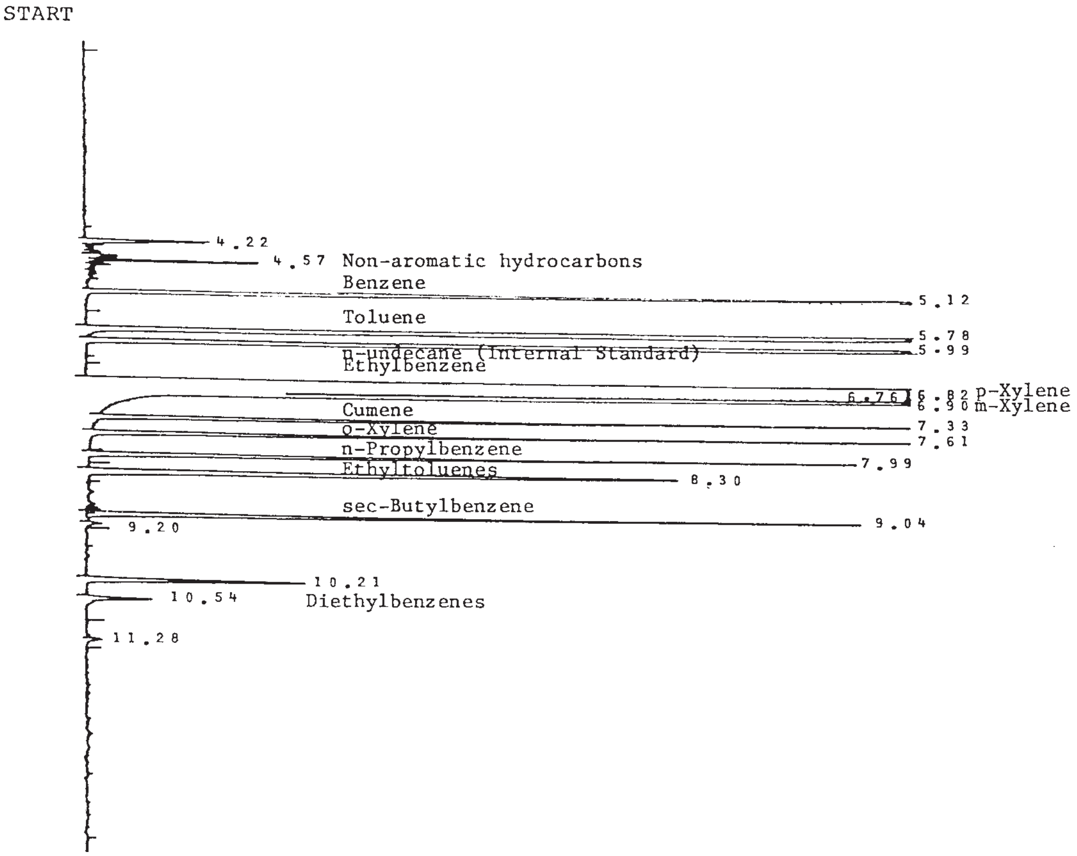

FIG. 1 Typical Chromatogram (see Table 1)

obtained by this test method. The criteria were derived from a round robin between seven laboratories. The data were obtained over two days using different operators.

13.1.1 *Intermediate Precision*—Results in the same laboratory should not be considered suspect unless they differ by more than the amount shown in Table 2.

13.1.2 *Reproducibility*—The results submitted by two laboratories should not be considered suspect unless they differ by more than the amount shown in Table 2.

13.2 *Bias*—No statement is made about bias since no acceptable reference material and value are available.

TABLE 2 Intermediate Precision and Reproducibility

Component	Concentration, Weight %	Intermediate Precision	Reproducibility
sec-Butylbenzene	0.002	0.001	0.003
n-Propylbenzene	0.010	0.002	0.003
m,p-Ethyltoluenes	0.014	0.003	0.002
o-Xylene	0.013	0.004	0.007
Cumene	0.012	0.003	0.002
Benzene	0.024	0.004	0.005
Toluene	0.592	0.083	0.100
m,p-Xylene	0.090	0.024	0.019
Diethylbenzenes	0.008	0.001	0.003
Ethylbenzene	99.05	0.200	0.186

14. Keywords

14.1 ethylbenzene; ethylbenzene purity; impurities in ethylbenzene

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