Designation: D5060 – 12

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

This standard is issued under the fixed designation D5060; 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 In determining the performance 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 a specific hazard statement, see Section 8.

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
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
E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

2.2 *Other Documents:*

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 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. Interferences

5.1 A key operational parameter for this method is the separation of *p*-xylene from ethylbenzene. Care should be taken during calibration to ensure the separation of these two components. If *p*-xylene is not separated from ethylbenzene during the calibration of the instrument, modify the column flow rate slightly until separation is achieved.

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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 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**

6. Apparatus

6.1 *Gas Chromatograph (GC),* any GC built for capillary column chromatography and equipped with a flame ionization detector (FID). The system shall have sufficient sensitivity, linearity, and range to obtain a minimum peak height response for 0.0010 wt% impurity of twice the height of the signal background noise, while not exceeding the full scale of either the detector or the electronic integration for the major components. It shall have a split injection system that will not discriminate over the boiling range of the samples analyzed.

6.2 *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.

6.3 *Chromatographic Data System* is required.

6.4 *Microsyringe,* 10-µL.

6.5 *Microsyringe,* 50-µL.

6.6 *Volumetric Flask,* 50-mL.

7. Reagents and Materials

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

7.2 *Compressed Air,* oil-free.

7.3 *Hydrogen,* chromatographic grade.

7.4 *Nitrogen,* chromatographic grade.

7.5 *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.

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

8. Hazards

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

9. Sampling

9.1 Guidelines for taking samples from bulk are given in Practice D3437.

10. Calibration

10.1 Prepare a calibration standard of ethylbenzene with impurities (listed in 7.5) at concentrations representative of those expected in the samples to be analyzed as described in Practice D4307.

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

10.3 Fill a 50-mL volumetric flask to the mark with ethylbenzene. With a microsyringe add 30 µL (or proper size) of the internal standard *n*-undecane. Mix well. The resulting stock ethylbenzene solution will contain 0.0512 weight % internal standard.

10.4 Analyze the calibration blend (10.2) as described in 11.3.

10.5 Analyze the stock ethylbenzene solution (10.3) as described in 11.3.

10.6 Calculate response factors as follows:

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

where:

R_i = response factor for impurity i relative to internal standard,
A_i = peak area of impurity i in calibration blend,
A_b = peak area of impurity i in stock ethylbenzene solution,
C_s = concentration of internal standard,
$A_{s,i}$ = peak area of internal standard in calibration blend,
$A_{s,b}$ = peak area of internal standard in stock ethylbenzene solution, and
C_i = concentration of impurity i, weight %.

10.7 Calculate response factor to the nearest 0.001.

11. Procedure

11.1 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 E355 and E1510.

11.2 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.

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

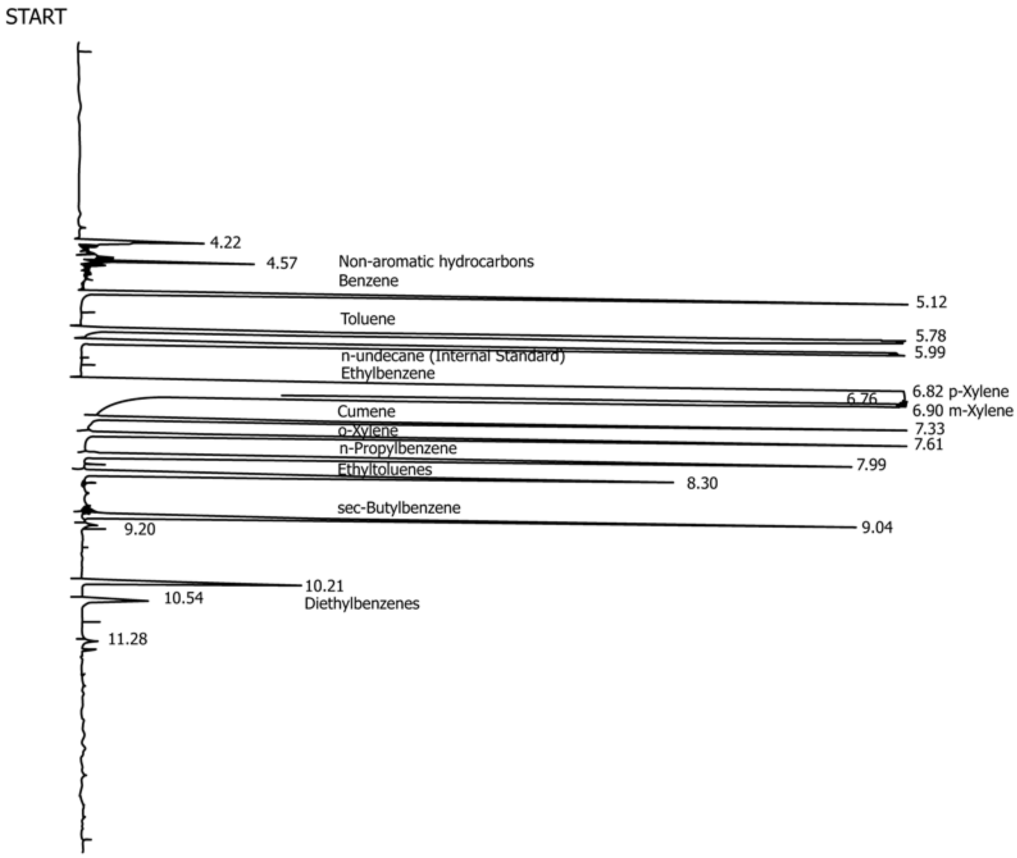

FIG. 1 Typical Chromatogram (see Table 1)

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

12. Calculation

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

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

12.3 Calculate the weight percent of the individual impurities, C_{i}, to the nearest 0.001 %, as follows:

$$C_{\mathrm{i}} = C_s \times A_i \times R_i / A_s \tag{2}$$

where:

C_s = concentration of internal standard, weight %,
A_i = peak area of impurity i,
R_i = response factor for impurity i, and
A_s = peak area of internal standard.

12.4 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.

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

13. Report

13.1 Report the following information:

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

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

14. Precision and Bias[4]

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

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

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

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

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1030. Contact ASTM Customer Service at service@astm.org.

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

15. Quality Guidelines

15.1 Laboratories shall have a quality control system in place.

15.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

15.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

15.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

15.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical quality control practices.

16. Keywords

16.1 ethylbenzene; ethylbenzene purity; impurities in ethylbenzene

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D5060 - 07) that may impact the use of this standard. (Approved June 1, 2012.)

(1) Added Section 1.4 on SI units.
(2) Replaced Section 6.3 recorder with chromatographic data system.
(3) Modified Section 10 on Calibration.
(4) Modified the definition of symbols in Equations 1 and 2.
(5) Modified Equation 2 in Section 12.
(6) Replaced Section 15 on Quality with the latest editorial guidelines.