Designation: D5060 – 07

StandardTest 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 *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.

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

[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**

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

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 blend of each compound listed in 7.5 and *n*-undecane at the 0.2 weight % level in ethylbenzene as described in Practice D4307. *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.

10.2 Analyze the ethylbenzene used in preparing the calibration blend as described in 11.3.

10.3 Analyze the calibration blend as described in 11.3.

10.4 Calculate response factors as follows:

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

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 %.

10.5 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.

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:

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