Designation: D7360 – 16

Standard Test Method for
Analysis of Benzene by Gas Chromatography with External Calibration[1]

This standard is issued under the fixed designation D7360; 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 normally occurring trace impurities in, and the purity of, finished benzene by gas chromatography with external calibration. A similar test method, using the internal standard technique of calibration is Test Method D4492.

1.2 This test method is applicable for nonaromatic hydrocarbon impurities at levels from 5 to 2000 mg/kg and for benzene purities of 99.80 weight % or higher.

1.3 This test method is applicable for aromatic impurities from 5 to 2000 mg/kg in benzene.

1.4 This test method has been found applicable to heteroatomic species such as 1,4-dioxane, from 10 to 2000 mg/kg in benzene.

1.5 The limit of detection for aromatic impurities is 0.9 mg/kg, 2.7 mg/kg for 1,4-dioxane and 1.1 mg/kg for methyl cyclohexane.

1.6 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.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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 9.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D3437 Practice for Sampling and Handling Liquid Cyclic Products
D3798 Test Method for Analysis of *p*-Xylene by Gas Chromatography (Withdrawn 2009)[3]
D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards
D4492 Test Method for Analysis of Benzene by Gas Chromatography
D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals
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
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E260 Practice for Packed Column Gas Chromatography
E355 Practice for Gas Chromatography Terms and Relationships
E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

2.2 *Other Document:*[4]
OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

3. Terminology

3.1 See Terminology D4790 for definition of terms used in this test method.

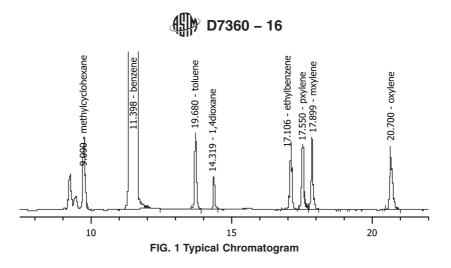[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.01 on Benzene, Toluene, Xylenes, Cyclohexane and Their Derivatives.
Current edition approved Nov. 1, 2016. Published January 2017. Originally approved in 2011. Last previous edition approved in 2011 as D7360 – 11. DOI: 10.1520/D7360-16.

[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] The last approved version of this historical standard is referenced on www.astm.org.
[4] 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

FIG. 1 Typical Chromatogram

TABLE 1 Typical Instrumental Parameters

Detector	flame ionization
Column:	fused silica
Length	50 m
Inside diameter	0.32 mm
Stationary phase	crosslinked polyethylene glycol
Film thickness	0.25 μm
Temperatures:	
Injector	200°C
Detector	250°C
Column	70°C isothermal
Carrier gas:	helium
Linear velocity	22 cm/s
Split ratio:	100:1
Sample size	2.0 μL
Recorder	electronic integration required

4. Summary of Test Method

4.1 A repeatable volume of the specimen to be analyzed is precisely injected into a gas chromatograph equipped with a flame ionization detector (FID) and a capillary column.

4.2 The peak area of each impurity is measured. The concentration of each impurity is determined using response factor or the linear calibration curve of peak area versus concentration. Purity is calculated by subtracting the sum of the impurities found from 100.00 weight %. The concentration of impurities are reported in mg/kg. Purity of benzene is reported in weight percent.

5. Significance and Use

5.1 This test method is suitable for determining the concentrations of known impurities in finished benzene and for use as an integral quality control tool where benzene is either produced or used in a manufacturing procedure. It is generally applied to impurities such as nonaromatics containing nine carbons or less, toluene, C8 aromatics, and 1,4-dioxane.

5.2 Absolute purity cannot be determined if undetected impurities are present.

6. Interferences

6.1 Benzene is typically resolved from components with boiling points <138°C that normally are found in purified benzene. Components normally found in purified benzene include nonaromatic hydrocarbons, toluene, C8 aromatics, and 1,4-dioxane. An adequate separation of known impurities from benzene should be evaluated for the column selected.

7. Apparatus

7.1 *Gas Chromatograph,* any chromatograph having a flame ionization detector that can be operated at the conditions given in Table 1.

7.2 *Electronic Integrator* chromatography data system.

7.3 *Column,* fused silica capillary column with cross-linked polyethylene glycol stationary phase is recommended. Alternate stationary phases may be used if they produce at least the same aromatic separation as achieved in the chromatogram in Fig. 1 and elute C_9 nonaromatic impurities before benzene.

7.4 *Automatic Injector*—The sample must be precisely and repeatably injected into the gas chromatograph. An automatic sample injection device is highly recommended.

8. Reagents and Materials

8.1 *Carrier Gas,* 99.999 % helium or hydrogen. Concentration of oxygen in the carrier should be <1 ppm, <0.5 ppm is preferred.

8.2 *Detector Gases, 99.999 %*—Nitrogen is recommended for make up 99.999 %. Hydrogen with THC <0.5 mg/kg. Air with CO2, CO and THC <1 mg/kg and oxygen between 20 and 22 %, is recommended.

8.3 *High Purity Benzene,* 99.999 weight % minimum, prepared by multiple step recrystallization of commercially available 99 + weight % benzene. See Annex A1. The benzene must be recrystalized a minimum of three times and then analyzed using this test method. Continue recrystalizing the benzene until no impurity greater than three times the noise level is detected.

8.4 Pure compounds for calibration should include toluene, *o*-, *m*-, and *p*-xylene, ethylbenzene, methylcyclohexane, and 1,4-dioxane of a purity not less than 99 %. If the purity of the calibration compounds is less than 99 %, the concentration and identification of impurities must be known so that the composition of the final weighed blends can be adjusted for the presence of the impurities.

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