Designation: D2360 – 08

Standard Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography[1]

This standard is issued under the fixed designation D2360; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method covers the determination of the total nonaromatic hydrocarbons, and trace monocyclic aromatic hydrocarbons in the purity of toluene and mixed xylenes by gas chromatography.

1.2 Nonaromatic aliphatic hydrocarbons containing 1 through 10 carbon atoms (methane through decanes) can be detected by this test method at concentrations ranging from 0.001 % to 2.500 weight %.

1.2.1 A small amount of benzene in mixed xylenes may not be distinguished from the nonaromatics and the concentrations are determined as a composite.

1.3 Monocyclic aromatic hydrocarbon impurities containing 6 through 9 carbon atoms (benzene through C_9 aromatics) can be detected by this test method at individual concentrations ranging from 0.001 % to 1.000 weight %.

1.4 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.

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

1.6 *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 statement, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D841 Specification for Nitration Grade Toluene

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D3797 Test Method for Analysis of *o*-Xylene by Gas Chromatography

D3798 Test Method for Analysis of *p*-Xylene by Gas Chromatography[3]

D4492 Test Method for Analysis of Benzene by Gas Chromatography

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

D5211 Specification for Xylenes for *p*-Xylene Feedstock

D6563 Test Method for Benzene, Toluene, Xylene (BTX) Concentrates Analysis by Gas Chromatography

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

E260 Practice for Packed Column Gas Chromatography

E355 Practice for Gas Chromatography Terms and Relationships

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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

[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.

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] Withdrawn.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

***A Summary of Changes section appears at the end of this standard.**

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

3. Terminology

3.1 *Definitions:*

3.1.1 For definition of terms used in this test method see Terminology D4790.

4. Summary of Test Method

4.1 A known amount of an internal standard is added to the specimen that is then introduced into a gas chromatograph (GC) 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 is calculated by subtracting the sum of the impurities found from 100.00. Results are reported either in weight percent or volume percent.

5. Significance and Use

5.1 The determination of hydrocarbon impurities contained in toluene and mixed xylenes used as chemical intermediates and solvents is typically required. This test is suitable for setting specifications and for use as an internal quality control tool where aromatic monocyclic hydrocarbons are produced or are used. This test method is applicable for determining the impurities from the aromatic hydrocarbon production process. Typical impurities are alkanes containing 1 to 10 carbon atoms, benzene, toluene, ethylbenzene (EB), xylenes, and aromatic hydrocarbons containing nine carbon atoms.

5.1.1 Refer to Test Methods D3797, D3798, and D4492 for determining the purity of *o*-Xylene, *p*-Xylene, and benzene, respectively.

5.1.2 Refer to Test Method D6563 for determining the C_8 aromatic hydrocarbon distribution in mixed xylenes.

5.2 Purity is commonly reported by subtracting the determined expected impurities from 100.00. However, a gas chromatographic analysis cannot determine absolute purity if unknown or undetected components are contained within the material being examined.

6. Interferences

6.1 The internal standard chosen must be satisfactorily resolved from any impurity and the product peak. A peak will be satisfactorily resolved from a neighboring peak if the distance from the valley to the baseline between the two peaks is not greater than 50 % of the peak height of the smaller of the two peaks.

6.2 In some cases for mixed xylenes, it may be difficult to resolve benzene from the nonaromatic hydrocarbons and therefore the concentrations are determined as a composite. In the event that the benzene concentration must be determined, an alternate method must be selected to ensure an accurate assessment of the benzene concentration.

TABLE 1 Instrumental Parameters

Detector	Flame ionization
Column:	
Tubing	fused silica
Stationary phase	crosslinked polyethylene glycol[A]
Film thickness, μ	0.25
Length, m	60
Diameter, mm	0.32 ID
Temperatures:	
Injector, °C	270
Detector, °C	300
Oven:	
Initial, °C	60
Time 1, min	10
Final, °C	150
Rate, °C/min	5
Time 2, min	10
Carrier gas	helium
Flow rate, mL/min	1.0
Split ratio	100:1
Sample size, μL	1.0
Analysis time, min	38
Linear velocity @ 145°C, cm/s	20

[A] Polyethylene glycol such as Carbowax 20 M available from most chromatographic suppliers, has been found suitable for this purpose.

7. Apparatus

7.1 *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 to background noise.

7.2 *Columns*—Both capillary and packed columns containing a stationary phase of cross-linked polyethylene glycol have been found satisfactory. The column must give satisfactory resolution of the internal standard from the solvent and the impurity peaks, and should be such that benzene is eluted between *n*-nonane and *n*-decane. Table 1 contains a description of a column that has been found satisfactory.

7.3 *Recorder*—Electronic integration is recommended.

7.4 *Microsyringe*, 10 and 50, and 500-μL capacity.

7.5 *Volumetric Flask*, 50-mL capacity.

8. Reagents

8.1 *Purity of Reagent*—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]

8.2 *Carrier Gas*—Helium is recommended. However, hydrogen may be used. Carrier, makeup and detector gases should have 99.999 % minimum purity. Oxygen in carrier gas should be less than 1 ppm; less than 0.5 ppm is preferred.

[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.

[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.