



Designation: D 2360 – 00

Standard Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography¹

This standard is issued under the fixed designation D 2360; 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 C₉ aromatics) can be detected by this test method at individual concentrations ranging from 0.001 % to 1.000 weight %.

1.4 The following applies to all specified limits in this test method: 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.

1.5 *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 specific hazard statement, see Section 9.

2. Referenced Documents

2.1 ASTM Standards:

D 841 Specification for Nitration Grade Toluene²

D 2306 Test Method for C₈ Hydrocarbon Analysis by Gas Chromatography²

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 June 10, 2000. Published August 2000. Originally published as D 2360 – 66 T. Last previous edition D 2360 – 95¹.

² Annual Book of ASTM Standards, Vol 06.04.

D 3437 Practice for Sampling and Handling Liquid Cyclic Products²

D 3797 Test Method for Analysis of *o*-Xylene by Gas Chromatography²

D 3798 Test Method for Analysis of *p*-Xylene by Gas Chromatography²

D 4492 Test Method for Analysis of Benzene by Gas Chromatography²

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals²

D 5211 Specification for Xylenes for *p*-Xylene Feedstock²

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³

E 260 Practice for Packed Column Gas Chromatography³

E 355 Practice for Gas Chromatography Terms and Relationships³

E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods³

E 1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs³

2.2 Other Document:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200⁴

3. Terminology

3.1 Definitions:

3.1.1 For definition of terms used in this test method see Terminology D 4790.

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.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington DC 20402.

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



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 D 3797, D 3798, and D 4492 for determining the purity of *o*-Xylene, *p*-Xylene, and benzene, respectively.

5.1.2 Refer to Test Method D 2306 for determining the C₈ 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.

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

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	30
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.

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*—Chromatographic grade helium is recommended.

8.3 *High Purity p-Xylene*, 99.999 weight % or greater purity.

8.3.1 Most *p*-xylene is available commercially at a purity less than 99.9 % and can be purified by recrystallization. To prepare 1.9 L of high purity *p*-xylene, begin with approximately 3.8 L of material and cool in an explosion-proof freezer at $-10 \pm 5^\circ\text{C}$ until approximately $\frac{1}{2}$ to $\frac{3}{4}$ of the *p*-xylene has frozen. This should require about 5 h. Remove the sample and decant the liquid portion. The solid portion is the purified *p*-xylene. Allow the *p*-xylene to thaw and repeat the crystallization step on the remaining sample until the *p*-xylene is free of contamination as indicated by gas chromatography.

8.4 *Pure Compounds*, for calibration, shall include *n*-nonane, benzene, toluene, ethylbenzene (EB), *o*-xylene and cumene. The purity of all reagents should be >99 weight %. If the purity is less than 99 %, the concentration and identification of impurities must be known so that the composition of the standard can be adjusted for the presence of the impurities.

8.4.1 *Internal Standard*—*n*-butylbenzene (NBB) is the recommended internal standard of choice, however, other compounds may be found acceptable provided they meet the criteria as defined in Section 6.

9. Hazards

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

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.

