



Designation: D7504 – 23

Standard Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography and Effective Carbon Number¹

This standard is issued under the fixed designation D7504; 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 total nonaromatic hydrocarbons and monocyclic aromatic hydrocarbons in benzene, toluene, ethylbenzene, *p*-xylene, *o*-xylene, styrene and mixed xylenes by gas chromatography. The purity of benzene, toluene, ethylbenzene, *p*-xylene, *o*-xylene, styrene and mixed xylenes is also calculated. Similar test methods, using the internal standard calibration technique and the external standard calibration technique, are Test Methods [D2360](#), [D5060](#), [D5135](#), and [D5917](#) respectively.

1.2 The limit of detection (LOD) is 0.0002 mass % and limit of quantitation (LOQ) is 0.0006 mass % for impurities in toluene, mixed xylenes, *p*-xylene, *o*-xylene, ethylbenzene, benzene, and styrene.

1.3 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](#).

1.4 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recom-*

mendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

- [D1555M](#) Test Method for Calculation of Volume and Weight of Industrial Aromatic Hydrocarbons and Cyclohexane [Metric]
- [D2360](#) Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography (Withdrawn 2016)³
- [D3437](#) Practice for Sampling and Handling Liquid Cyclic Products
- [D4790](#) Terminology of Aromatic Hydrocarbons and Related Chemicals
- [D5060](#) Test Method for Determining Impurities in High-Purity Ethylbenzene by Gas Chromatography
- [D5135](#) Test Method for Analysis of Styrene by Capillary Gas Chromatography
- [D5136](#) Specification for High Purity *p*-Xylene
- [D5211](#) Specification for Xylenes for *p*-Xylene Feedstock
- [D5917](#) Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography and External Calibration
- [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

¹ This test method is under the jurisdiction of ASTM Committee [D16](#) on Aromatic, Industrial, Specialty 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 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

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

2.2 Other Document:

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200⁴

3. Terminology

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

4. Summary of Test Method

4.1 The specimen to be analyzed is injected into a gas chromatograph equipped with a flame ionization detector (FID) and a capillary column. The peak area of each component is measured and adjusted using effective carbon number (ECN)⁵ correction factors. The concentration of each component is calculated based on its relative percentages of total adjusted peak area and normalized to 100.0000 %.

5. Significance and Use

5.1 Determining the type and amount of hydrocarbon impurities remaining from the manufacture of toluene, mixed xylenes, *p*-xylene, *o*-xylene, ethylbenzene, benzene, and styrene used as chemical intermediates and solvents is often required. This test method is suitable for setting specifications and for use as an internal quality control tool where these products are produced or are used. Typical impurities are: alkanes containing 1 to 10 carbons atoms, benzene, toluene, ethylbenzene (EB), xylenes, and aromatic hydrocarbons containing nine carbon atoms or more.

5.2 This method may not detect all components and there may be unknown components that would be assigned inappropriate correction factors and thus, the results may not be absolute.

6. Interferences

6.1 The complete separation of *p*-xylene from ethylbenzene, or ethylbenzene and *m*-xylene from *p*-xylene can be difficult when either ethylbenzene or *p*-xylene is analyzed, respectively. The separation can be considered adequate if the distance from the baseline to the valley between the two peaks is not greater than 50 % of the peak height of lower of the two peaks.

7. Apparatus

7.1 Chromatographic data system is required.

7.2 Columns—The choice of column is based on resolution requirements. Any column may be used that is capable of resolving all significant impurities from the major component. The column and conditions described in Table 1 have been

TABLE 1 Recommended Method Parameters

Inlet	Split
Temperature, °C	270
Column:	
Tubing	fused silica
Length, m	60
Internal diameter, mm	0.32
Stationary phase	crosslinked polyethylene glycol
Film thickness, µm	0.25
Column temperature program	
Initial temperature, °C	60
Initial time, min	10
Programming rate, °C/min	5
Final, °C	150
Time 2, min	10
Carrier gas	helium or hydrogen
Linear velocity, cm/s at 145 °C	20 helium or 45 hydrogen
Split ratio	100:1
Sample size, µL	0.6
Detector:	flame ionization
Temperature, °C	300
Analysis time, min	38

used successfully and shall be used as a referee in cases of dispute. A 60 m column with an internal diameter of 0.32 mm and a film thickness of 0.50 µm crosslinked polyethylene glycol may improve resolution.

7.3 *Gas Chromatograph*—Any instrument having a flame ionization detector and a splitter injector suitable for use with a fused silica capillary column may be used, provided the system has sufficient sensitivity, linearity, and range to determine 0.0001 mass %, while not exceeding the full scale of either the detector or the electronic integration for the major component. It shall have a split injection system that will not discriminate over the boiling range of the samples analyzed. The system should be capable of operating at conditions given in Table 1.

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

7.5 *Syringe*—Chromatographic, capable of delivering appropriate µL volumes.

8. Reagents and Materials

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,⁶ where such specifications are available. Reagents with an establish purity greater than ACS reagent grade may be used.

8.1.1 Alpha-methylstyrene (AMS) CAS 98-83-9,

8.1.2 Benzene CAS 71-43-2,

8.1.3 Cumene (isopropylbenzene) CAS 98-82-8,

8.1.4 1,4-dioxane CAS 123-91-1,

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 Scanlon, J. T. and Willis, D. E., "Calculation of Flame Ionization Detector Relative Response Factors Using the Effective Carbon Number Concept," *Journal of Chromatographic Science*, Vol. 23, August 1985, pp. 333–339.

⁶ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.