



Standard Test Method for C₈ Aromatic Hydrocarbon Analysis by Gas Chromatography¹

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1. Scope

1.1 This test method determines the relative distribution of the individual C₈ aromatic hydrocarbon isomers in the following xylene products:

1.1.1 Nitration grade xylene conforming to Specification D 843.

1.1.2 Xylenes for *p*-Xylene feedstock conforming to Specification D 5211.

1.1.3 Ten-degree xylene conforming to Specification D 846.

1.2 The absolute concentration of hydrocarbon impurities typically found in commercially available mixed C₈ aromatic hydrocarbons should be determined using Test Method D 2360.

1.3 The following applies to all specified limits in this test method: for purposes of determining conformance with this test method, 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 ASTM Practice E 29.

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 specific hazard statements, see Section 9.

2. Referenced Documents

2.1 ASTM Standards:

D 843 Specification for Nitration Grade Xylene²

D 846 Specification for Ten-Degree Xylene³

D 2360 Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography²

D 3437 Practice for Sampling and Handling Liquid Cyclic Products²

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 *Annual Book of ASTM Standards*, Vol 06.04.

³ Discontinued 1991. See *1990 Annual Book of ASTM Standards*, Vol 06.03.

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 4626 Practice for Calculation of Gas Chromatographic Response Factors⁴

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals²

D 5060 Test Method for Determining Impurities in High-Purity Ethylbenzene by Gas Chromatography²

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

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁵

E 260 Practice for Packed Column Gas Chromatography⁵

E 355 Practice for Gas Chromatography Terms and Relationships⁵

E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods⁵

2.2 Other Document:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200⁶

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 The specimen is introduced into a gas chromatograph equipped with either a flame ionization detector (FID) or thermal conductivity detector (TCD). Either packed or capillary columns are permitted. The peak area of each component is measured and the weight percent concentration is calculated by dividing the peak area of the individual component by the sum of the total individual peak areas. The result is multiplied by 100 to get the normalized C₈ aromatic hydrocarbon isomer distribution.

⁴ *Annual Book of ASTM Standards*, Vol 05.02.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

⁶ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

5. Significance and Use

5.1 This test method is suitable for setting specifications on the materials referenced in 1.1.1 through 1.1.3. This test method may also be used as an internal quality control tool and in development or research work.

5.1.1 Refer to Methods D 5060, D 3797, and D 3798 to determine the purity of ethylbenzene, *o*-xylene, and *p*-xylene respectively.

5.2 This test method does not attempt to determine the absolute purity of the sample, but defines the relative distribution of the C₈ aromatic hydrocarbons.

6. Interferences

6.1 If present, nonaromatic hydrocarbons of twelve carbons or greater will be interferences in this analysis.

7. Apparatus

7.1 *Gas Chromatograph*—Any instrument having a flame ionization detector or a thermal conductivity detector may be used. A flame ionization detector is preferred.

7.2 *Columns*—Both capillary and packed columns containing a stationary phase of cross-linked polyethylene glycol have been found satisfactory. The column and instrumental conditions described in Table 1 are recommended.

7.3 *Recorder*—Electronic integration is recommended.

8. Reagents

8.1 *Carrier Gas*—Chromatographic grade hydrogen, helium or nitrogen have been found acceptable.

9. Hazards

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

10. Sampling

10.1 Sample the material in accordance with Practice D 3437.

TABLE 1 Instrumental Parameters

	Column A
Detector	flame ionization
Column:	
Tubing	fused silica
Stationary phase	polyethylene glycol
Film thickness, μ	0.25
Length, m	50
Inside diameter, mm	0.25
Temperatures:	
Injector, °C	200
Detector, °C	250
Oven:	
Initial, °C	70
Time 1, min	10
Final, °C	120
Rate, °C/min	5
Time 2, min	0
Carrier gas	helium
Linear Vel., cm/s	20
Split ratio	100:1
Sample size, μL	1.0
Analysis time, min	20

11. Preparation of Apparatus

11.1 The method used to prepare packed columns is not critical provided that the finished column produces the desired separation.

11.2 Follow manufacturer's instructions for mounting and conditioning the column into the chromatograph and adjusting the instrument to the conditions described in Table 1. Allow sufficient time for the equipment to reach equilibrium. See Practices E 260 and E 355 for additional information on gas chromatography practices and terminology.

11.3 The column must be capable of resolving the C₈ aromatic hydrocarbons as individual isomers.

11.3.1 The resolution of two components is defined as follows:

$$R = \frac{2(t_{R,m} - t_{R,p})}{(w_{b,m} + w_{b,p})} \quad (1)$$

where:

- $t_{R,m}$ = > $t_{R,p}$
- R = peak resolution,
- $t_{R,m}$ = retention time of *m*-xylene, (m = *m*-xylene),
- $t_{R,p}$ = retention time of *p*-xylene, (p = *p*-xylene),
- $w_{b,m}$ = peak width at baseline for *m*-xylene, and
- $w_{b,p}$ = peak width at baseline for *p*-xylene.

11.4 For *p*-xylene and *m*-xylene, the minimal allowable resolution is 1.0. If the resolution between *p*-xylene and *m*-xylene is <1.0, the chromatographic system must be modified to improve the separation.

12. Procedure

12.1 Inject the desired volume of specimen into the gas chromatograph and record the peaks on the sensitivity setting that allows the maximum peak height and minimum baseline noise. The injection volume should be small enough to produce symmetrical gaussian shaped peaks. Fig. 1 illustrates a typical analysis.

13. Calculations

13.1 Determine the area defined by each peak.

13.1.1 In the event that a thermal conductivity detector is used for the analysis, the observed peak areas may need to be adjusted using response factors. See Practice D 4626 for a discussion on response factors.

13.2 Calculate the weight percent concentration of each C₈ aromatic hydrocarbon as follows:

$$C_i = \frac{(A_i)(100)}{(A_s)} \quad (2)$$

where:

- C_i = concentration of component i,
- A_i = peak area of component i, and
- A_s = peak area of all C₈ aromatic isomers.

14. Report

14.1 Report the individual C₈ aromatic hydrocarbons to the nearest 0.01 weight %.