Designation: D 2306 – 00

Standard Test Method for C_8 Aromatic Hydrocarbon Analysis by Gas Chromatography[1]

1. Scope

1.1 This test method determines the relative distribution of the individual C_8 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 C_8 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 Xylene[2]

D 846 Specification for Ten-Degree Xylene[3]

D 2360 Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography[2]

D 3437 Practice for Sampling and Handling Liquid Cyclic Products[2]

D 3797 Test Method for Analysis of *o*-Xylene by Gas Chromatography[2]

D 3798 Test Method for Analysis of *p*-Xylene by Gas Chromatography[2]

D 4626 Practice for Calculation of Gas Chromatographic Response Factors[4]

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals[2]

D 5060 Test Method for Determining Impurities in High-Purity Ethylbenzene by Gas Chromatography[2]

D 5211 Specification for Xylenes for *p*-Xylene Feedstock[2]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[5]

E 260 Practice for Packed Column Gas Chromatography[5]

E 355 Practice for Gas Chromatography Terms and Relationships[5]

E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods[5]

2.2 *Other Document:*

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200[6]

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 C_8 aromatic hydrocarbon isomer distribution.

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[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.

[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 C_8 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 C_8 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 C_8 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 C_8 aromatic isomers.

14. Report

14.1 Report the individual C_8 aromatic hydrocarbons to the nearest 0.01 weight %.

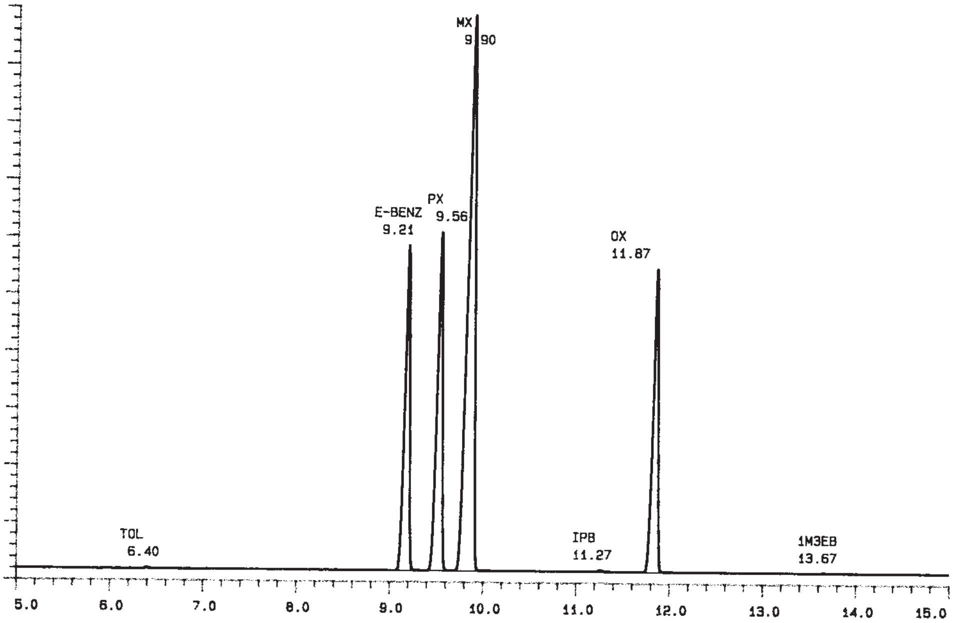

FIG. 1 Chromatogram of Mixed Xylenes Using Conditions for Column A, Table 1

15. Precision and Bias [7]

15.1 The following criteria should be used to judge acceptability (95 % probability level) of results obtained by this test method. The criteria were derived from an interlaboratory test amongst 10 laboratories. The data were run on 2 days using different operators using a sample that was gravimetrically prepared from the individual C_8 aromatic hydrocarbons to the concentrations listed in Table 2. Fused silica capillary columns containing a polyethylene glycol stationary phase were used by 9/10 laboratories. Flame ionization detection was used by all 10 laboratories. The results of the round robin were analyzed in accordance with Practice E 691.

TABLE 2 Intermediate Precision (formerly called Repeatability) and Reproducibility

Component	Expected Concentration Weight%	Average Concentration Reported Weight %	Intermediate Precision (formerly called Repeatability	Reproducibility
Ethylbenzene	17.93	17.92	0.077	0.153
p-Xylene	20.16	20.07	0.073	0.193
m-Xylene	44.09	43.96	0.101	0.369
o-Xylene	17.83	17.98	0.116	0.193

15.1.1 *Intermediate Precision (formerly called Repeatability)*—Results in the same laboratory should not be considered suspect unless they differ by more than the amount for repeatability shown in Table 2. On the basis of test error alone, the difference between two test results obtained in the same laboratory on the same material will be expected to exceed this value only about 5 % of the time.

15.1.2 *Reproducibility*—Results submitted by each of two laboratories should not be considered suspect unless they differ by more than the amount shown for reproducibility in Table 2. On the basis of test error alone, the difference between two test results obtained in different laboratories on the same material will be expected to exceed this value only about 5 % of the time.

15.2 *Bias*—Although the interlaboratory test utilized a sample prepared gravimetrically from individual C_8 isomers obtained at the highest purity available, this sample has not been approved as an acceptable reference material and consequently bias has not been determined.

15.2.1 As an aid for the users in determining the possibility of bias, calculated C_8 distributed for the round robin sample is listed in Table 2 as the "Expected Concentration." The average value for each C_8 isomer as calculated from the reported concentrations is listed as "Average Concentration Reported."

16. Keywords

16.1 aromatic hydrocarbon; aromatic hydrocarbon analysis; C_8; gas chromatography; isomer;*p*-Xylene; xylene

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[7] Supporting data are available from ASTM International Headquarters. Request RR: D16-1015.

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