Designation: D 3798 – 03

Standard Test Method for
Analysis of *p*-Xylene by Gas Chromatography[1]

This standard is issued under the fixed designation D 3798; 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 known hydrocarbon impurities in, and the purity of *p*-xylene by gas chromatography (GC). It is generally meant for the analysis of *p*-xylene of 99 % or greater purity. Impurity concentrations that can be measured range from 0.001 to 1.000 weight %.

1.2 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 right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

1.3 *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 a specific hazard statement, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

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

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

D 5136 Specification for High Purity *p*-Xylene[2]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[3]

E 260 Practice for Packed Column Gas Chromatography[3]

E 355 Practice for Gas Chromatography Terms and Relationships[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

E 1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs[4]

2.2 *Other Document:*

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200[5]

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 a specimen of *p*-xylene. The prepared specimen is mixed and analyzed by a gas chromatograph equipped with a flame ionization detector (FID). The peak area of each impurity and the internal standard is measured. 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 (the *p*-xylene content) is calculated by subtracting the sum of the impurities found from 100.00. Results are reported in weight percent.

5. Significance and Use

5.1 This test method is suitable for setting specifications on *p*-xylene and for use as an internal quality control tool where *p*-xylene is produced or is used in a manufacturing process. It may also be used in development or research work involving *p*-xylene. It is generally applied to determining those commonly occurring impurities such as nonaromatic hydrocarbons, benzene, toluene, ethylbenzene, *m*-xylene, *o*-xylene, and cumene (isopropylbenzene).

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 components are contained within the material being examined. Refer to Specification D 5136 for determining other chemical and physical properties of *p*-xylene.

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

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[5] 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.**

6. Interferences

6.1 The internal standard chosen must be sufficiently resolved from any impurity and the *p*-xylene peak. Refer to 8.4.1.

7. Apparatus

7.1 *Gas Chromatograph*—Any chromatograph 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 a 0.001 weight % impurity twice the height of the signal background noise.

7.2 *Columns*—Different columns have been found satisfactory, depending on the purity of the *p*-xylene to be analyzed.

7.2.1 *p-Xylene Range from 99.0 to 99.8 %*—Both capillary and packed columns have been found satisfactory. The column must give satisfactory separation of the internal standard from *p*-xylene and the impurity peaks. Complete separation of ethylbenzene and *m*-xylene from *p*-xylene is difficult and can be considered adequate if the distance from the baseline to the valley between peaks is not greater than 50 % of the peak height of the impurity. Table 1 contains a description of two columns that have been found satisfactory.

7.2.2 *p-Xylene Range 99.8 % and Greater*—Only capillary columns have been found satisfactory. Complete separation of ethylbenzene and *m*-xylene from *p*-xylene is difficult and can be considered adequate if the distance from the baseline to the valley between peaks is not greater than 50 % of the peak height of the impurity. It is important that tangential skimming is employed. Table 1 contains a description of one column that has been found satisfactory.

7.3 *Recorder*—optional

7.4 Electronic integration is required. Tangent skimming capabilities are required because of the difficulty in fully separating impurities from *p*-xylene.

7.5 *Microsyringe*, 100-µL capacity.

7.6 *Volumetric Flask*, 100-mL capacity.

TABLE 1 Typical Instrument Conditions for *p*-Xylene Analysis

	A	B
Column:		
Tubing	fused silica	stainless steel
Stationary phase	crosslinked	diisodecylphthalate Bentone 34[A]
Concentration, weight %	polyethylene glycol	3.5 %/3.5 %
Solid support	not applicable	flux-calcined diatomite[B]
Mesh size	not applicable	60 to 80
Film thickness, µ	0.25	not applicable
Length, m	50	6.1
Inside diameter, mm	0.32	3.2
Carrier gas:	helium	helium
Flow rate, mL/min	1.0	30
Split ratio	100:1	not applicable
Temperature, °C:		
Inlet	200	200
Dectector	200	200
Column	60	60
Internal Standard	*n*-undecane	*n*-octane

[A] The sole source of supply of the material known to the committee at this time is Bentone 34, available from National Lead Co. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

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.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *High Purity p-Xylene* (99.99 % or greater purity)—Most *p*-xylene is available commercially at a purity less than 99.9 weight %, but can be purified by recrystallization. To prepare 2 qt of high-purity *p*-xylene, begin with approximately 1 gal of reagent-grade *p*-xylene and cool in an explosion-proof freezer at – 10± 10°C until approximately ½ to ¾ of the *p*-xylene has frozen. This should require about 5 h. Remove the sample and decant the liquid portion. 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.

8.3 *Carrier Gas*—Chromatographic-grade nitrogen, helium, or hydrogen have been found satisfactory for the *p*-xylene range from 99.0 to 99.8 %. However, only helium or hydrogen have been found satisfactory for the *p*-xylene range of 99.8 % and greater.

8.4 *Pure Compounds for Calibration*, shall include *m*-xylene, *o*-xylene, toluene, ethylbenzene, isopropylbenzene (cumene), and *n*-nonane. The purity of all reagents should be 99 % or greater. 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*-Undecane (NC11) is the recommended internal standard of choice for Conditions *A* and *n*-octane (NC8) for Conditions B in Table 1. However, other compounds may be found acceptable provided they meet the criteria as defined in Section 6 and 8.4.

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.

10. Sampling

10.1 Sample the material in accordance with Practice D 3437.

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.

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[6] *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.