Designation: D3797 – 05

StandardTest Method for
Analysis of *o*-Xylene by Gas Chromatography[1]

This standard is issued under the fixed designation D3797; 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 analysis of normally occurring impurities in, and the purity of, *o*-xylene by gas chromatography. Impurities determined include nonaromatic hydrocarbons, benzene, toluene, *p*- and *m*-xylenes, cumene, styrene, and ethylbenzene.

1.2 This test method is applicable for impurities at concentrations from 0.001 to 2.000 % and for *o*-xylene purities of 98 % or higher.

1.3 The following applies to all specified limits in this standard: 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 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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

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

E355 Practice for Gas Chromatography Terms and Relationships

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

2.2 *Other Documents:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200[3]

3. Terminology

3.1 *Definitions:*

3.1.1 For definition of terms used in this test method see Terminology D4790.

4. Summary of Test Method

4.1 A known amount of internal standard is added to the sample. A gas chromatograph equipped with a flame ionization detector and a polar-fused silica capillary column is used for the analysis. The impurities are measured relative to the internal standard. To calculate *o*-Xylene purity subtract the impurities found from 100.00 %.

5. Significance and Use

5.1 This test method is suitable for setting specifications on *o*-xylene and for use as an internal quality control tool where *o*-xylene is used in a manufacturing process. It may be used in development or research work involving *o*-xylene.

5.2 Purity is commonly reported by subtracting the determined expected impurities from 100 %. Absolute purity cannot be determined if unknown impurities are present.

6. Apparatus

6.1 *Gas Chromatography*—Any gas chromatograph having a flame ionization detector and a splitter injector suitable for

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

use with a fused-silica capillary column may be used, provided the system has sufficient sensitivity to obtain a minimum peak height response of 0.1 mV for 0.010 % internal standard when operated at the stated conditions. Background noise at these conditions is not to exceed 3 µV.

6.2 *Chromatographic Column,* fused silica capillary, 60-m long, 0.32-mm inside diameter, internally coated to a 0.5-µm thickness with a bonded (cross-linked) polyethylene glycol. Other columns may be used after it has been established that such column is capable of separating all major impurities and the internal standard from the *o*-xylene under operating conditions appropriate for the column.

6.3 *Recorder,* optional.

6.4 Electronic integration with tangent capabilities (required).

6.5 *Microsyringes,* 10-µL, and 50-µL.

6.6 *Volumetric Flask,* 50-mL.

7. Reagents and Materials

7.1 *Carrier Gas,* hydrogen or helium, 99.999 % minimum purity. Oxygen content in carrier less than 1 ppm (less than 0.5 ppm preferred). Purify to remove hydrocarbons and water.

7.2 *Compressed Air,* 99.999 % minimum purity. Purify to remove hyrdocarbons and water.

7.3 *Hydrogen,* 99.999 % minimum purity. Purify to remove hydrocarbons, oxygen, and water.

7.4 *Nitrogen,* 99.999 % minimum purity. Purify to remove hydrocarbons, oxygen, and water.

7.5 Pure compounds for calibration shall include *n*-nonane (Note 1), toluene, styrene, ethylbenzene, *p*-xylene, *m*-xylene, *o*-xylene, cumene (isopropylbenzene), *iso*-octane (2,2,4-trimethylbenzene), (Note 2), and *n*-undecane (Note 2), of a purity not less than 99 %. If the purity of the calibration compounds is less than 99 %, the concentration and identification of impurities must be known so that the composition of the final weighed blends can be adjusted for presence of the impurities.

NOTE 1—*n*-nonane represents the nonaromatic in a sample.
NOTE 2—Any of these compounds may be used as an internal standard provided it elutes free of contamination.

8. Hazards

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

9. Sampling

9.1 Guidelines for taking samples from bulk are given in Practice D3437.

10. Calibration

10.1 Prepare a synthetic mixture in accordance with Practice D4307 from pure hydrocarbons with all of the aromatic compounds present in the sample to be analyzed containing approximately 98.0 weight % *o*-xylene and the expected significant impurities at their expected concentration. A typical composition is given in weight percent. *o*-Xylene of a purity not less than 99 weight % must be used in preparing the calibration mixture. The pure material itself must be analyzed and corrections made in the composition of the calibration blend as required. Sample the material in accordance with Practice D3437. The standard may be purchased if desired.

Typical Concentrations for Calibration Blend

o-xylene	98.25 %
toluene	0.2 %
p-xylene	0.2 %
m-xylene	0.4 %
ethylbenzene	0.2 %
cumene	0.3 %
n-nonane	0.2 %
styrene	0.05 %

10.2 Analyze the *o*-xylene used in preparing the calibration blend as described in 11.2 and 11.3. Calculate the purity of the stock *o*-xylene as shown in 12.3, using an assumed response factor of 1.00 for each impurity. This will verify that the *o*-xylene used in preparing this test method is 99 weight % or better.

10.3 Analyze the calibration blend as described in 11.3.

10.4 Calculate response factors as follows:

$$R_i = \frac{C_i}{(C_s)\left(\frac{A_i}{A_{si}} - \frac{A_b}{A_{sb}}\right)} \qquad (1)$$

where:
R_i = response factor for impurity relative to internal standard,
A_i = area of impurity peak in calibration blend,
A_b = area of impurity in the stock *o*-xylene,
C_s = concentration of internal standard, weight %,
A_{si} = area of internal standard peak in calibration blend,
A_{sb} = area of internal standard peak in stock *o*-xylene, and
C_i = concentration of impurity, weight, %.

10.5 Calculate response factors to the nearest 0.001.

11. Procedure

11.1 Install the chromatograph column and establish stable instrument operation at the operating conditions shown in Table 1 (Note 1). Refer to instructions provided by the manufacturer of the gas chromatograph and Practices E355 and E1510.

11.2 Fill a 50-mL volumetric flask to the mark with test specimen. With a microsyringe, add 30 µL of the internal standard. Mix well. When using *iso*-octane as the internal standard and assuming densities of 0.692 for *iso*-octane and 0.880 for *o*-xylene, the resulting *iso*-octane concentration will be 0.0472 weight %. When using *n*-undecane as the internal standard and assuming densities of 0.740 for *n*-undecane and 0.880 for *o*-xylene, the resulting *n*-undecane concentration will be 0.0504 weight %.

11.3 Inject between 0.5 to 1.0 µL of solution into the gas chromatograph and obtain the chromatogram. A typical chromatogram is shown in Fig. 1. An injection size of 0.6 µL was used for Fig. 1 and for the intermediate precision described in Section 14 and Table 2.