Designation: D 6526 – 00

Standard Test Method for Analysis of Toluene by Capillary Column Gas Chromatography[1]

This standard is issued under the fixed designation D 6526; 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 hydrocarbon impurities typically found in, and the purity of, samples containing 98 wt % and greater toluene. This test method is applicable to impurity concentrations in the range of 0.0010 to 0.500 wt %.

1.2 Monocyclic aromatic hydrocarbons containing 6 through 8 carbon atoms, cumene, 1,4–dioxane, and nonaromatic aliphatic hydrocarbons containing up to 12 carbon atoms can be detected by this test method. The nonaromatic compounds are determined as a composite.

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

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

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

2.2 *Other Document:*

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200[4]

3. Terminology

3.1 See Terminology D 4790 for definitions of terms used in this test method.

4. Summary of Test Method

4.1 A portion of the sample is injected into a gas chromatograph using a microlitre syringe at the specified conditions of the test method. The toluene and other components are separated as they are transported through the column by an inert carrier gas. The components in the effluent are measured by a flame ionization detector (FID). The area of the impurity peaks and toluene are electronically integrated. The peak areas are corrected with effective carbon number (ECN)[5] response factors and normalized to 100.0000 %.

5. Significance and Use

5.1 This test method is suitable for determining the concentrations of known impurities in refined toluene and for use as an integral quality control tool where toluene is produced or used in manufacturing.

5.2 Toluene purity is reported, but a chromatographic analysis cannot determine absolute purity if unknown or undetected components are present in the sample.

6. Interferences

6.1 If present, nonaromatic hydrocarbons of 13 carbons or greater, alcohols, ethers, and other similar organic compounds can interfere with this test method by co-eluting with the aromatic hydrocarbons.

6.2 Compounds not detected by a FID are not determined by this test method.

6.3 Nonvolatile material is not determined.

---

[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.

Current edition approved Feb. 10, 2000. Published April 2000.

[2] *Annual Book of ASTM Standards*, Vol 06.04.

[3] *Annual Book of ASTM Standards*, Vol 014.02.

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

[5] Scanlon, J. T., and Willis, D. E., "Calculation of Ionization Detector Relative Response Factors Using the Effective Carbon Number Concept", *Journal of Chromatographic Science*, Vol 35, August, 1985, pp. 333-339.

7. Apparatus

7.1 *Gas Chromatograph (GC)*—any GC built for capillary column chromatography. The system shall have sufficient sensitivity, linearity, and range to obtain a minimum peak height response for 0.0010 wt % impurity of twice the height of the signal background noise, 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.

TABLE 1 Typical Instrumental Parameters

Detector:	Flame ionization
Detector temperature, °C	150°C
Column:	50 m by 0.25 mm
Tubing	Fused silica
Stationary phase	TCEP
Film thickness, µm	0.40
Column temperature, °C	70
Carrier Gas	Helium
Linear velocity at 70°C, cm/s	25
Inlet:	Split
Injection port temperature, °C	150°C
Split ratio	40
Split flow, mL/min	55
Sample size, µL	1.0

7.2 *Recorder*—electronic integration is recommended.

7.3 *Capillary Column*—fused silica capillary column with 1,2,3-tris-2-cyano-ethoxypropane (TCEP) phase is recommended. Other columns may be used after it has been established that such a column is capable of separating all major impurities under operating conditions appropriate for the column.

7.4 *Microsyringe*—capable of delivering 1 µL of sample.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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, unless otherwise indicated. 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 *Carrier Gas*—Helium, 99.99 mole % minimum, is recommended.

8.3 *FID Detector Gases*:

8.3.1 *Hydrogen*—99.99 mole % minimum.

8.3.2 *Air*—less than 10 ppm each of total hydrocarbons and water.

9. Hazards

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

10. Sample Handling

10.1 Collect the samples in accordance with Practice D 3437.

10.2 To preserve sample integrity (consistency) and prevent the loss of volatile components, which may be in some samples, do not uncover samples any longer than necessary.

11. Preparation of Apparatus

11.1 Follow the manufacturer's instructions for mounting and conditioning the column in the chromatograph.

11.2 Adjust the instrument to the conditions as described in Table 1 to give the proper separations. Allow sufficient time for the instrument to reach equilibrium as indicated by a stable baseline. See Practices E 355 and E 1510 for additional information on gas chromatography practices and terminology.

12. Procedure

12.1 Inject an appropriate amount of specimen, typically 1.0 µL, into the chromatograph. A low purity toluene sample chromatogram, which shows the relative retention time of components typically found in commercial toluene, is illustrated in Fig. 1.

NOTE 1—Since TCEP is a nonbonded phase, significant retention time shifts can occur with column condition.

12.2 Measure the area of all peaks. The nonaromatics fraction includes all peaks eluting before benzene. Sum together all nonaromatic peaks and report as a total area.

13. Calculation

13.1 Using the ECN weight response factors listed in Table 2, calculate the concentration of each component as follows:

$$C_i = 100 \times (A_i \times R_i) / \sum_{i=1}^{n} (A_i \times R_i) \quad (1)$$

where:

C_i = concentration results for component(s) *i*, weight %,
A_i = peak area of component(s) *i*, and
R_i = ECN response factor for component(s) *i*.

14. Report

14.1 Report the following information:

14.1.1 Report impurity concentrations less than 0.0010 % as <0.0010 %.

14.1.2 Report greater than 0.0010 % nonaromatics, benzene, ethylbenzene, xylenes, and cumene to the nearest 0.0001 %.

14.1.3 Report toluene purity to the nearest 0.01 %.

15. Precision and Bias

15.1 *Precision*—The following criteria should be used to judge the acceptability of results obtained by this test method (95 % confidence level). The precision criteria were derived from one laboratory performing ten analyses on three standards during a two-day period. The results of the precision study were calculated using Practice E 691.

[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.

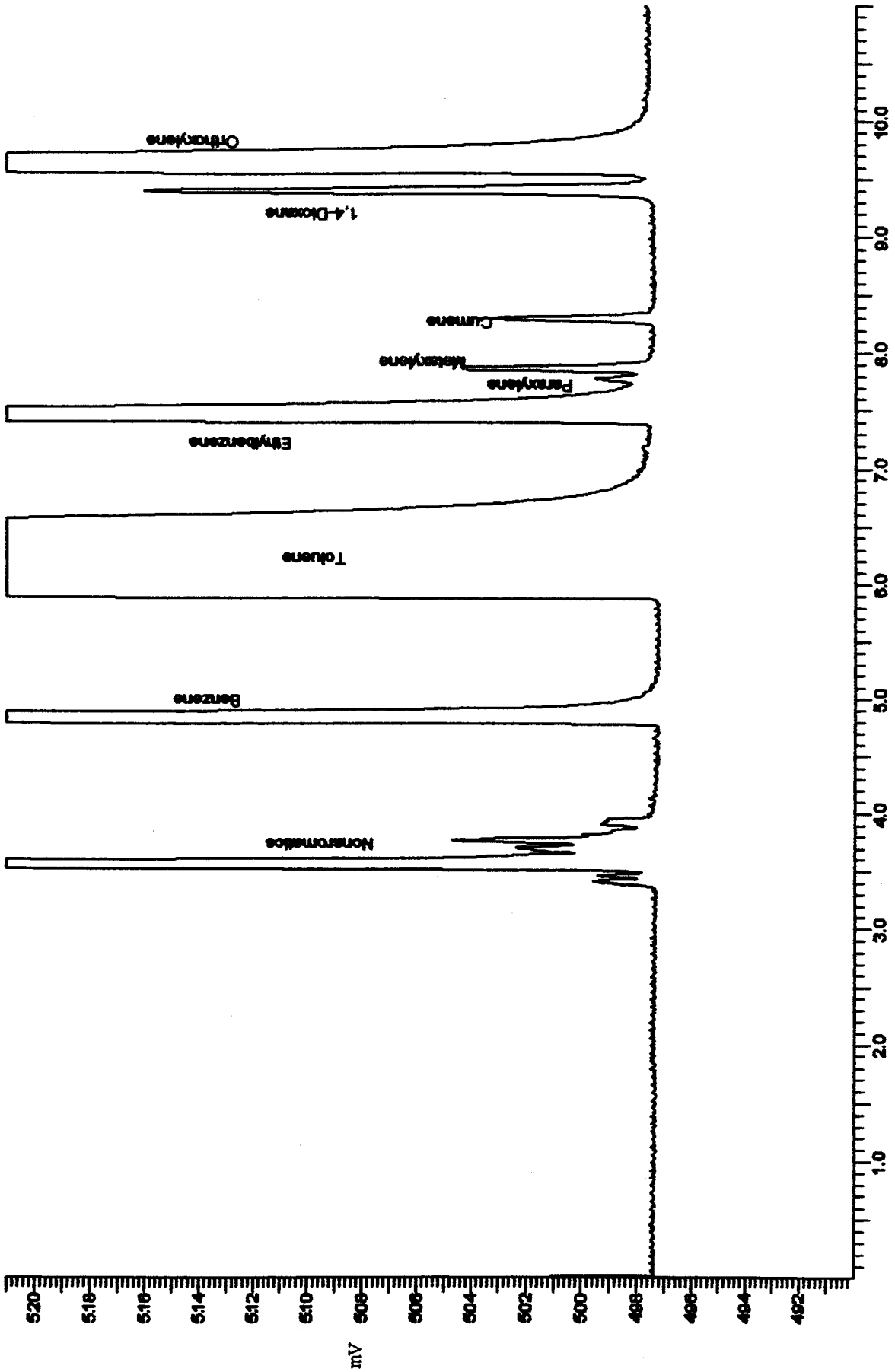

FIG. 1 Low Purity Toluene Sample Chromatogram

TABLE 2 Effective Carbon Number Response Factors

Component	Response Factor (Weight)
Nonaromatics:	0.9975
Benzene	0.9100
Toluene	0.9200
Ethylbenzene	0.9275
p-Xylene	0.9275
m-Xylene	0.9275
o-Xylene	0.9275
Cumene	0.9333
1,4-Dioxane	3.0800

15.1.1 *Intermediate Precision (formerly called Repeatability)*—Duplicated results by the same operator should not be considered suspect unless they differ by more than the amount shown in Table 3.

15.1.2 *Reproducibility*—The reproducibility of this test method has not been determined.

15.1.3 *Bias*—Since there was no accepted reference material available at the time of testing, no statement on bias can be made at this time.

TABLE 3 Intermediate Precision

Compound	Concentration (Weight Percent)	Repeatability (Weight Percent)
n-Octane	0.0211	0.0006
	0.3038	0.0020
	0.5078	0.0017
Benzene	0.0074	0.0003
	0.0561	0.0008
	0.5104	0.0056
Ethylbenzene	0.0150	0.0003
	0.2986	0.0031
	0.4963	0.0031
1,4-Dioxane	0.0010	0.0003
	0.0203	0.0006
	0.0000	0.0000
p-Xylene	0.0147	0.0003
	0.2945	0.0034
	0.4979	0.0031
Toluene	99.9400	0.0006
	99.0300	0.0067
	97.9900	0.0028

16. Keywords

16.1 benzene; gas chromatography; impurities;1,4-dioxane; toluene purity