



Designation: D 6563 – 00

Test Method for Benzene, Toluene, Xylene, (BTX) Concentrates Analysis by Capillary Column Gas Chromatography¹

This standard is issued under the fixed designation D 6563; 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 the total nonaromatic hydrocarbons, benzene, toluene, ethylbenzene, xylenes, and C₉ + aromatic hydrocarbons in BTX concentrates by capillary column gas chromatography. This test method is applicable to materials with a final boiling point below 215°C.

1.2 Individual components can be determined from 0.01 to 90 %.

1.3 The following applies to all specified limits in this standard: for purpose of determining conformance with this standard, 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.

1.4 *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 a specific precautionary statement, see Section 9.

2. Referenced Documents

2.1 ASTM Standards:

D 3437 Practice for sampling and Handling Liquid Cyclic Products²

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³

E 355 Practice for Gas Chromatography Terms and Relationships³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

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.

E 1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs³

2.2 Other Documents:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200⁴

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *extracted reformate*—An aromatic concentrate obtained by solvent extraction of reformate.

3.1.2 *reformate*—The product of a catalytic process that increases the concentration of aromatic hydrocarbons.

3.1.3 *pyrolysis gasoline*—Depentanized by-product recovered from ethylene manufacture.

3.1.4 *synthetic blend*—Blend of reagent hydrocarbons that simulate a process product.

3.1.5 *hydrogenated pyrolysis gasoline*—Pyrolysis gasoline that has been treated with hydrogen to reduce the olefins content.

3.1.6 *crude ethylbenzene*—Product produced from the reaction of impure fluid cat cracking, (FCC) ethylene and benzene.

3.1.6.1 *Discussion*—It typically contains greater than 40 % of ethylbenzene and benzene.

3.1.7 *light blending aromatics feedstock*—Light aromatics fraction (with high amounts of benzene and toluene) typically recovered from the isomerization of a *p*-xylene or *m*-xylene depleted C₈ aromatics stream.

4. Summary of Test Method

4.1 The specimen to be analyzed is injected into a gas chromatograph equipped with a flame ionization detector (FID) and a capillary column. The peak area of each component is measured and adjusted using effective carbon number (ECN) response factors. The concentration of each component is calculated based on its relative percentages of total adjusted

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

peak area and normalized to 100.00. Results can be reported as either volume or weight percent.

4.2 Volumetric results can be derived by dividing each component's weight percent by its relative density and re-normalizing to 100 %.

5. Significance and Use

5.1 This test method was primarily developed to determine benzene, toluene, and xylenes in chemical intermediate and solvent streams such as reformat, BTX extracts, pyrolysis gasoline, hydrogenated pyrolysis gasoline, crude benzene, crude ethylbenzene, commercial toluene, and light blending aromatic feedstocks. This test method may not detect all components and there may be unknown components that would be assigned inappropriate response factors and thus, the results may not be absolute.

6. Interferences

6.1 Nonaromatic hydrocarbons may interfere with the determination of benzene and toluene when certain columns are used.

6.2 Styrene may be present in some samples. It will elute with C₉+ aromatics.

7. Apparatus

7.1 *Gas Chromatograph*—Any gas chromatograph having a flame ionization detector and a splitter injector suitable for 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 0.01 % of any component of interest in the sample being analyzed or in the system blend when operated at the stated conditions. Background noise should not exceed 0.01 mV. Largest component of interest should not exceed 90 % full scale of the electronic integration device.

7.2 *Column*—Capillary columns have found to be satisfactory. For example, a 60 m by 0.25 mm inside diameter fused silica capillary, internally coated to a 0.25- μ m thickness with a bonded (cross-linked) polyethylene glycol can be used (see [Table 1](#) for parameters). 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.

7.3 *Recorder/Electronic Integration*—Electronic integration with tangent capabilities is recommended.

8. Reagents

8.1 *Carrier Gas*—Helium with a minimum purity of 99.99 mol %.

8.2 *Detector Gas*—Hydrogen with a minimum purity of 99.99 mol %.

8.3 *Flame Support Gas*—Air, hydrocarbon free, with a minimum purity of 99.99 mol %.

TABLE 1 Instrument Parameters

Column	Size of 60 m by 0.25 mm ID μ -bonded polyethylene glycol-fused silica capillary, internally coated to a 0.25- μ m thickness
Carrier gas	helium
Flow, linear velocity at 70°C, cm/s	20
Split ratio	200:1
Detector gas	
Hydrogen flow rate, mL/min	30
Air flow rate, mL/min	300
Make-up flow rate, mL/min	30
Sample size, μ L	0.5
Temperatures	
Injector, °C	250
Detector, °C	300
Column	
Initial, °C	70
Hold, min	10
Rate, °C/min	5
Final, °C	200
Hold, min	24

9. Hazards

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

10. Sampling

10.1 Sample material in accordance with Practice [D 3437](#).

11. Preparation of Apparatus

11.1 *Chromatograph*—Follow manufacturer's instructions for mounting and conditioning the column in the chromatograph and adjusting the instrument to the conditions as described in [Table 1](#) to give the desired separation. Allow sufficient time for the instrument to reach equilibrium as indicated by a stable recorder/electronic baseline. See Practices [E 355](#) and [E 1510](#) for additional information on gas chromatography practices and terminology.

12. Procedure

12.1 Bring the sample to ambient room temperature.

12.2 Inject an appropriate amount of sample into the chromatograph that meets the criteria outlined in [7.1](#). See Practices [E 355](#) and [E 1510](#) for additional information on gas chromatography practices and terminology.

12.3 Sample chromatograms are illustrated in [Figs. 1-3](#).

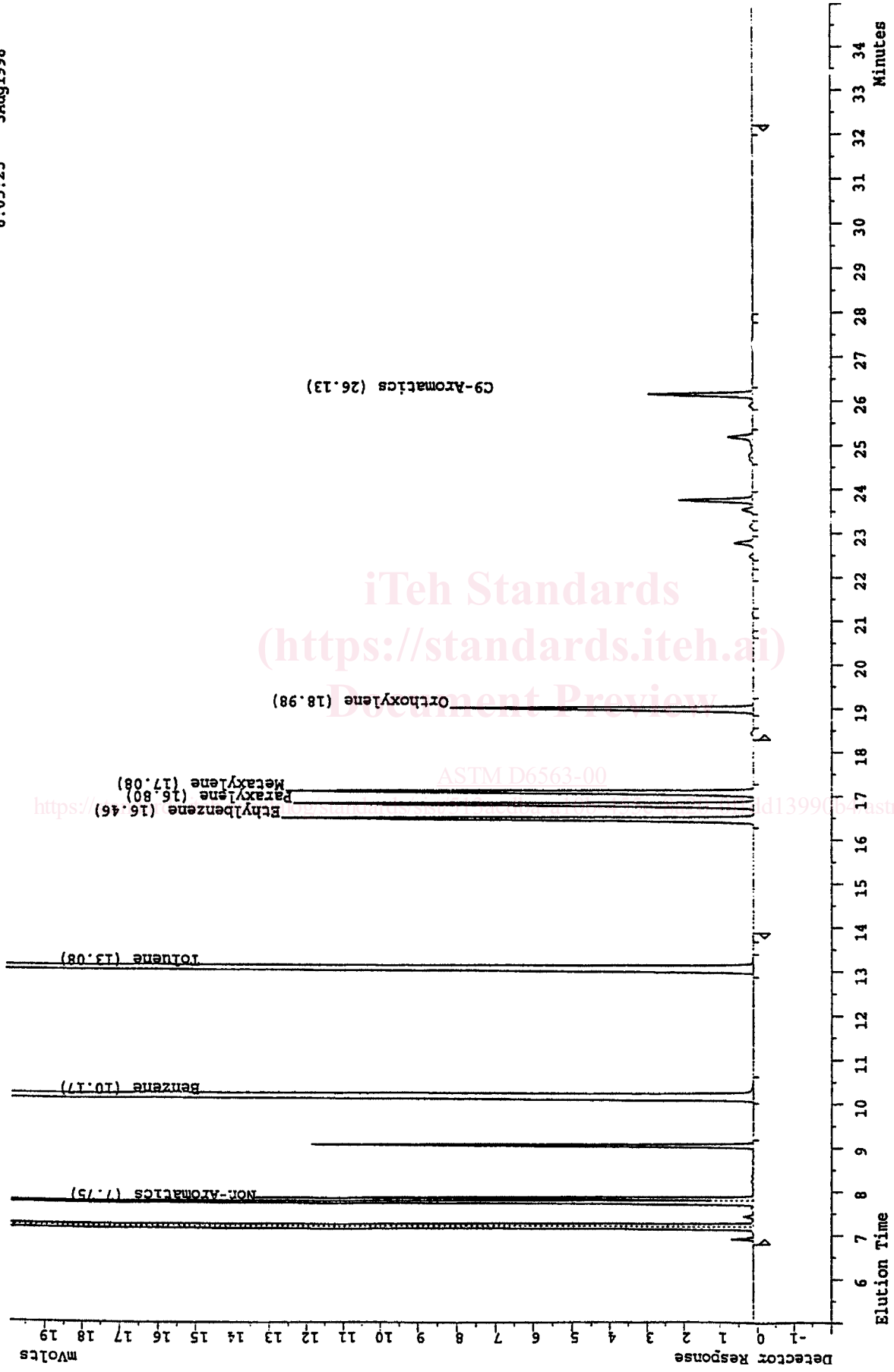
12.4 Measure the area of all peaks. The nonaromatics fraction includes all peaks up to ethylbenzene (except for the peaks assigned to benzene and toluene). Sum together all the nonaromatic peaks as a total area. The C₉+ aromatics fraction includes cumene and all peaks eluting after *o*-xylene. Sum together all the C₉+ aromatic peaks as a total area.

13. Calculation

13.1 Calculate the weight percent concentration of each component as follows:

$$C_i = \frac{A_i \times ECN_i}{\sum_{i=1}^n (A_i \times ECN_i)} \quad (1)$$

8:05:25 3Aug1998



Inject time: 14:26:01 27Jul1998 Vial: 0

Instrument: 026
FIG. 1 Synthetic Blend

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GC2263

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