Standard Test Method for
Benzene, Toluene, Xylene (BTX) Concentrates Analysis by Gas Chromatography[1]

This standard is issued under the fixed designation D6563; 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 total C_9 + 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 This test method may also be used to determine the relative distribution of the individual C_8 aromatic hydrocarbon isomers in mixed xylenes.

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

1.4 In determining the conformance of the test results using this method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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:*[2]

D3437 Practice for Sampling and Handling Liquid Cyclic Products

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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 of Terms Specific to This Standard:*

3.1.1 *extracted reformate, n*—an aromatic concentrate obtained by solvent extraction of reformate.

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

3.1.3 *pyrolysis gasoline, n*—depentanized by-product recovered from ethylene manufacture.

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

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

3.1.6 *crude ethylbenzene, n*—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, n*—light aromatics fraction (with high amounts of benzene and toluene) typically recovered from the isomerization of a *p*-xylene or *m*-xylene depleted C_8 aromatics stream.

3.1.8 *mixed xylenes, n*—a mixture of C_8 aromatic hydrocarbon isomers including ethylbenzene, but excluding stryene.

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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] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

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

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 peak area and normalized to 100.00. To determine the relative distribution of C_8 aromatic hydrocarbons, the peak areas of those components only are normalized to 100.00.

4.2 Results can be reported as either volume or weight percent. 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 reformate, 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.

5.2 The relative distribution of C_8 aromatics is useful for determining conformance to *p*-xylene feedstock specifications.

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 C_9+ 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, linearity, and range to obtain a minimum peak height response for a 0.01 % peak of five times the height of the signal background noise, while not exceeding the full scale of either the detector or the electronic integration for the highest peak. The split injection system shall not discriminate over the boiling range of the samples analyzed. The system shall be capable of operating at the conditions given in Table 1.

7.2 *Columns*—The choice of column is based upon resolution requirements. Any column may be used that is capable of resolving all the components of interest. The column and conditions described in Table 1 have been used successfully and will be the referee in case of dispute.

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 %. See 11.1.

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

8.3 *Flame Support Gas*—Air, total, hydrocarbon less than 5 ppm.

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 D3437.

TABLE 1 Instrument Parameters

Column	50 or 60 m by 0.25 mm ID bonded polyethylene glycol-fused silica capillary, internally coated to a 0.25-µm 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, µL	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

11. Preparation of Apparatus

11.1 *Chromatograph*—Follow manufacturer's instructions for mounting and conditioning the column in the chromatograph. Adjust the instrument to the conditions as described in Table 1 to give the desired separation using the suggested column. Other columns ~~may require different conditions~~and conditions, including the use of hydrogen carrier gas, may be used to achieve the resolution requirements. Allow sufficient time for the instrument to reach equilibrium as indicated by a stable recorder/electronic baseline. See Practices E355 and E1510 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 E355 and E1510 for additional information on gas chromatography practices and terminology.

12.3 Sample chromatograms are illustrated in Figs. 1-4.

12.4 Measure the area of all peaks. The non-aromatics fraction includes all peaks up to ethylbenzene (except for the peaks assigned to benzene and toluene). Sum together all the non-aromatic peaks as a total area. The C_9+ aromatics fraction includes all peaks eluting after *m*-xylene except for *o*-xylene. Sum together all the C_9+ aromatic peaks as a total area.

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ASTM D6563-11a
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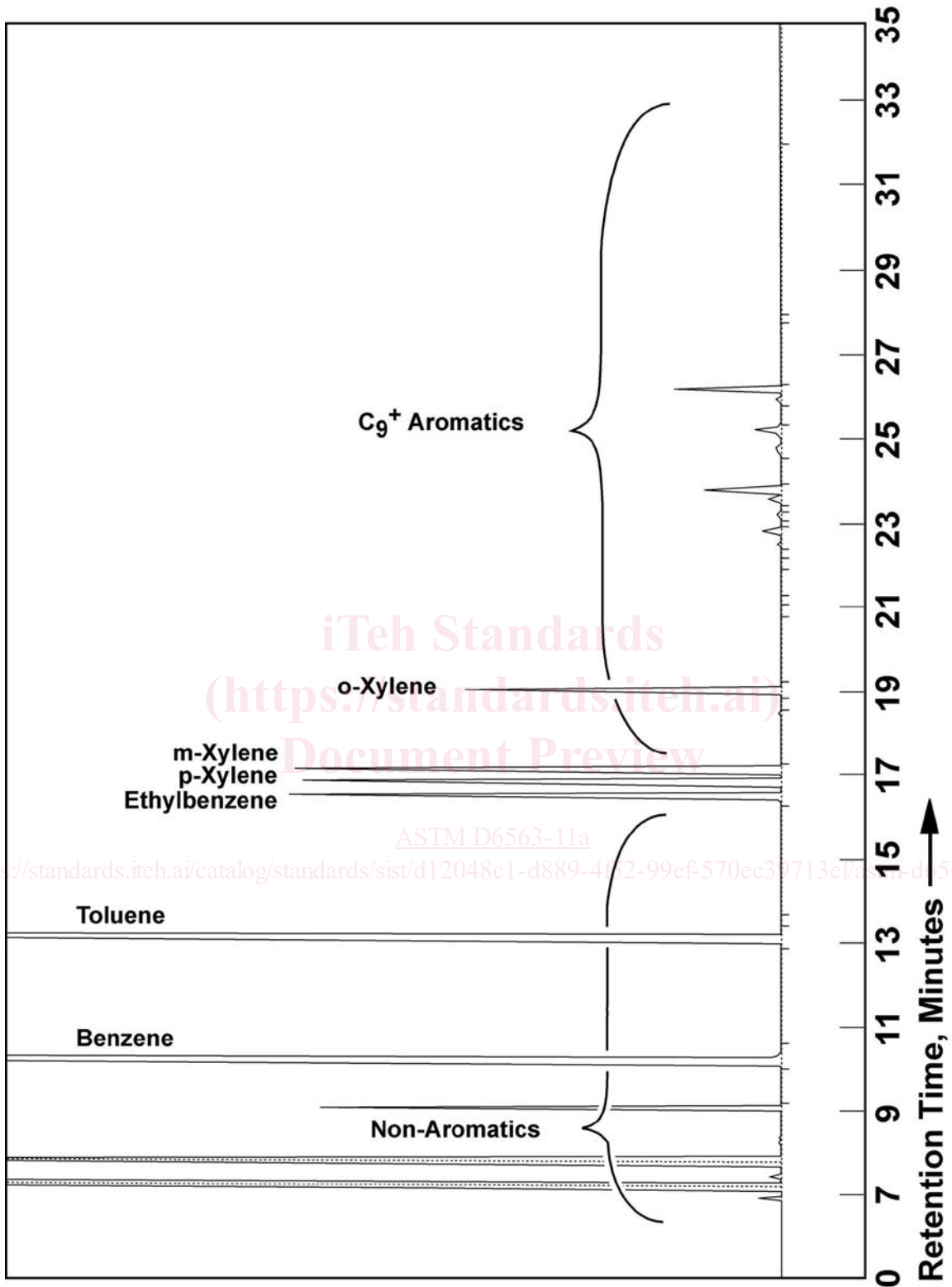
FIG. 1 Synthetic Blend

13. Calculation

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

$$C_i = \frac{100 \times A_i \times ECN_i}{\sum_{i=1}^{n} (A_i \times ECN_i)}$$

(1)
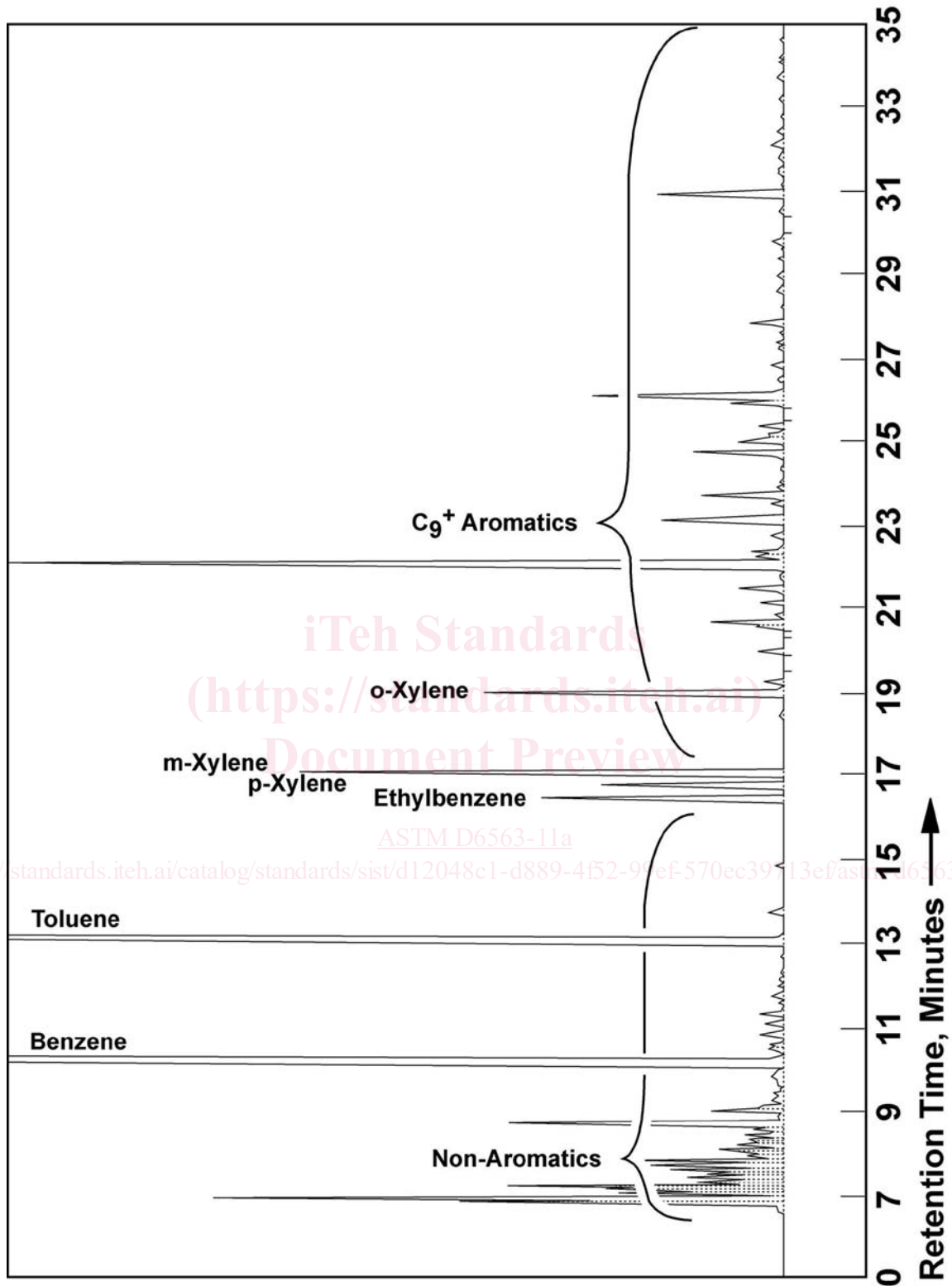
4

FIG. 2 Pyrolysis Gasoline

where:

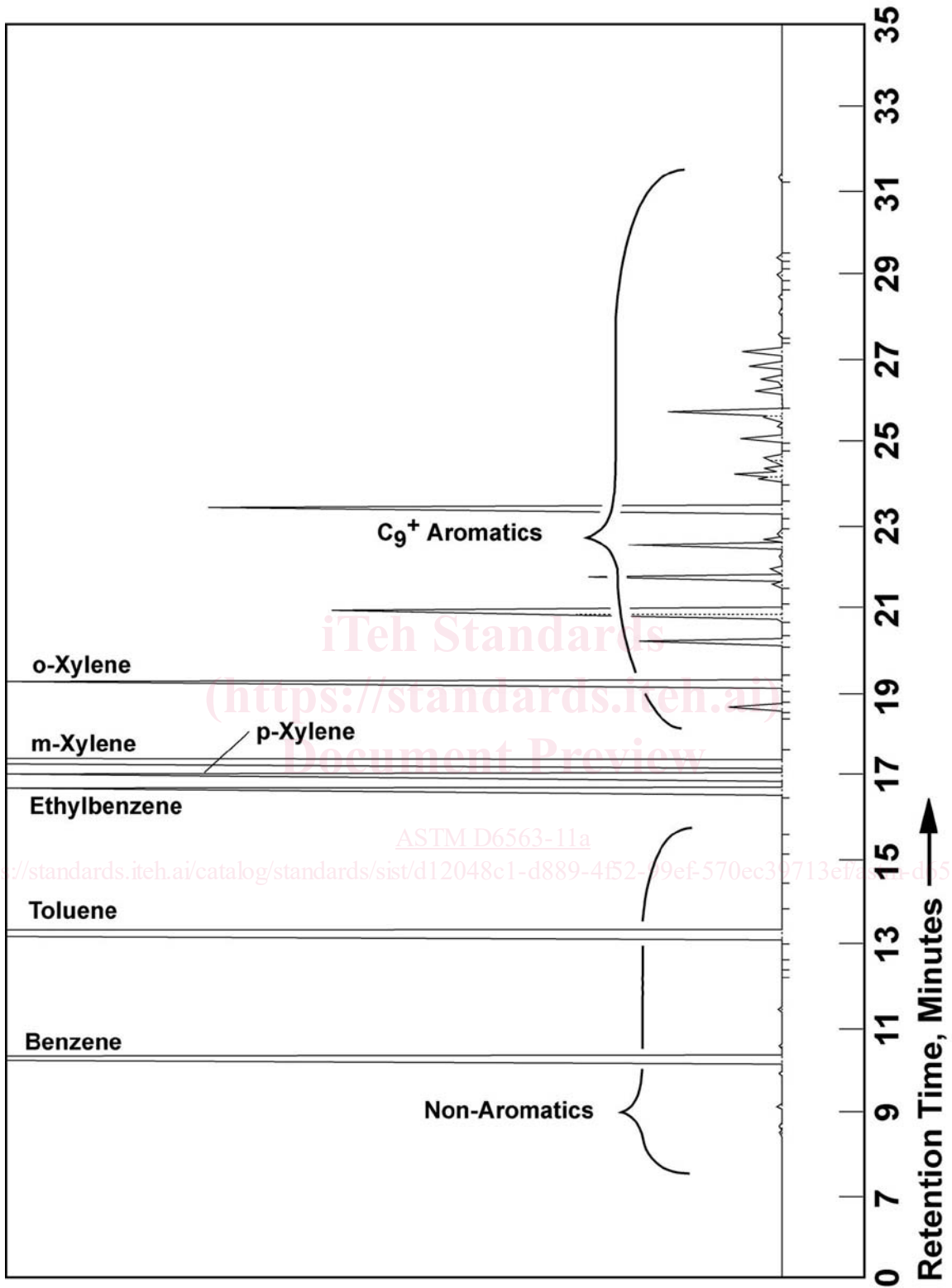|	
C_i	= concentration of component in weight percent,
A_i	= area of component, i peak,
ECN_i	= effective carbon response factor for component, and
$\sum_{i=1}^{n}(A_i \times ECN_i)$	= the summation of all response corrected areas in the chromatogram.

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FIG. 3 Extracted Reformate

13.2 Calculate the volume percent concentration of each component as follows:

$$V = \frac{100 \; C/D}{\sum_{i=1}^{n} (C/D)} \qquad (2)$$