



Designation: D3257 – 22

Standard Test Methods for Aromatics in Mineral Spirits by Gas Chromatography¹

This standard is issued under the fixed designation D3257; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the determination of ethylbenzene and total eight-carbon (C_8) and heavier aromatics in the concentration range from 0.1 to 30 % in mineral spirits having a distillation range from 149 to 210 °C (300 to 410 °F) as determined by Test Method D86. The procedures permit the identification and calculation of concentrations of aromatic components to 0.1 volume %.

1.2 It is recognized by analytical chemists that a single column gas chromatography analysis of an unknown sample is risky. In such cases, multiple and different analytical techniques must be used for absolutely positive identification, for example, several different gas chromatography columns, gas chromatography/mass spectrometer, or gas chromatography/infrared, etc. In these test methods the material is known and is clearly defined.

1.3 Oxygenated compounds, if present, may interfere and cause erroneous results. Such oxygenated compounds are not normally present in mineral spirits.

1.4 Three test methods are covered as follows:

1.4.1 *Test Method A*, measurement of ethylbenzene content, C_8 plus higher aromatics (except ethylbenzene), and total aromatics by means of a single packed column gas chromatographic analysis.

1.4.2 *Test Method B*, measurement of ethylbenzene content by means of a rapid packed column gas chromatographic analysis.

1.4.3 *Test Method C*, measurement of ethylbenzene content, C_8 plus higher aromatics (except ethylbenzene) and total aromatics by means of a capillary column gas chromatographic analysis.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 For purposes of determining conformance of an observed or a calculated value using this test method to relevant specifications, test result(s) 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.

1.7 For hazard information and guidance, see the supplier’s Material Safety Data Sheet.

1.8 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards*:²

D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³

E260 Practice for Packed Column Gas Chromatography

2.2 *ASTM Adjuncts*:

D2PP Determination of Precision and Bias Data⁴

¹ These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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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 The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from ASTM International Headquarters. Order Adjunct No. ADJD6300.

3. Summary of Test Methods

3.1 The material, with an internal standard, is introduced into a gas chromatographic column containing a strongly polar liquid phase. The polar phase has very little affinity for saturated and olefinic hydrocarbons while exhibiting a pronounced retention of aromatics. This selectivity, which is illustrated in Fig. 1 results in the elution of all saturated and olefinic hydrocarbons in the products described above prior to the elution of toluene. Either a thermal conductivity or flame ionization detector may be used. Calibration is obtained in Test Method A and C from a synthetic blend of the most important aromatic compounds. Internal standards are used in all three test methods. Typical chromatograms are shown in Fig. 2 and Fig. 3.

NOTE 1—Refer to Practice E260 for additional information on gas chromatography techniques.

4. Significance and Use

4.1 These test methods were developed to measure the types and amounts of aromatics in mineral spirits to determine compliance with air pollution regulations that restrict the aromatic content of solvents. They have been demonstrated to be workable and to produce accurate results. However, due to the sensitivity of the tests to operating variables, some labora-

tories having limited experience with gas chromatographic analyses of hydrocarbons may experience difficulty in performing the tests.

TEST METHOD A—ETHYLBENZENE AND TOTAL AROMATICS CONTENTS BY MEANS OF A SINGLE PACKED COLUMN GAS CHROMATOGRAPHIC ANALYSIS

5. Apparatus

5.1 *Chromatograph*, any gas chromatographic instrument that has the following performance and characteristics:

5.1.1 *Sensitivity*—The overall sensitivity must be sufficient to detect 0.1 volume % of any aromatic compound of interest with a peak height of at least 10 % of full-scale chart deflection without loss of resolution as defined in 5.1.2, or 10 times the noise level.

5.1.2 *Column*—Any column and conditions may be used provided the system meets all the following criteria when the test blend is injected into the chromatograph and the chromatogram recorded in accordance with 7.2, is analyzed as follows:

5.1.2.1 Construct tangents to the peak to intersect the baseline for the *n*-tridecane (*n*C₁₃) and toluene peaks. Measure the distance between the two peaks and the width of each peak

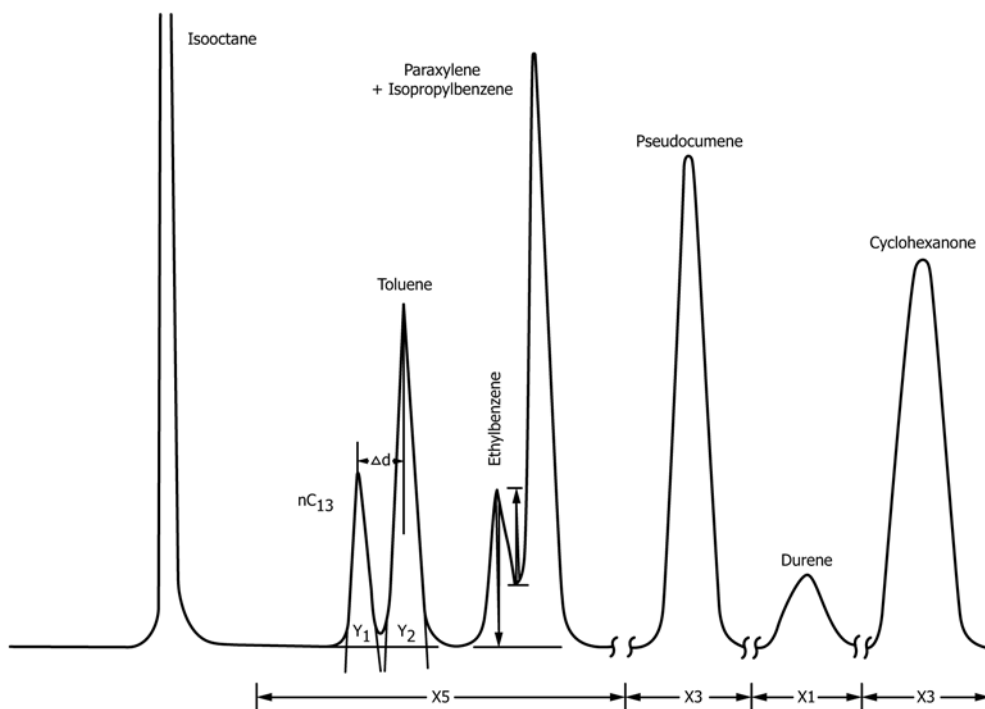


FIG. 1 Typical Resolution of Test Blend Using Packed Column

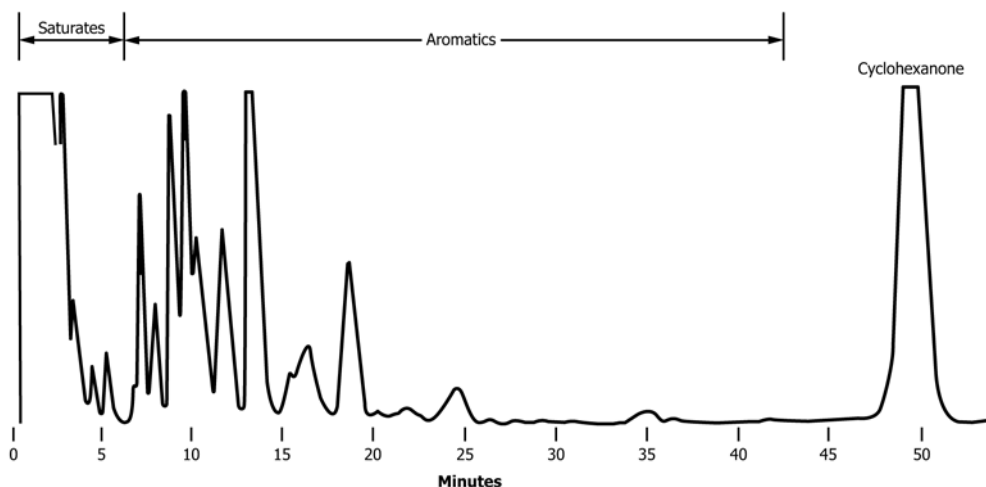


FIG. 2 Typical Packed Column Chromatogram of Mineral Spirits (Column and Conditions Described in Table 2)

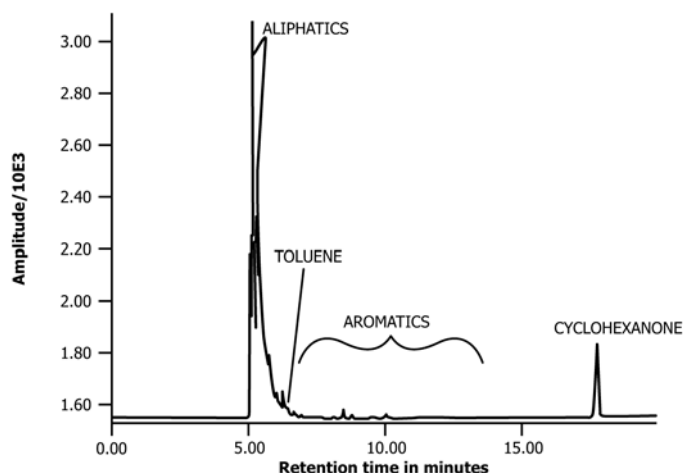


FIG. 3 Typical Capillary Chromatogram of Mineral Spirits (Column and Conditions Described in Table 3)

as the distance along the baseline under the peak between the points of intersection (see Fig. 1).

5.1.2.2 Calculate the peak resolution,

$$R = 2\Delta d / (Y_1 + Y_2) \quad (1)$$

where:

- Δd = distance between nC_{13} and toluene peaks,
- Y_1 = width of nC_{13} peak along the baseline, and
- Y_2 = width of the toluene peak along the baseline.

The peak resolution, R , must be not less than 0.9, otherwise the resolution of ethylbenzene may be lost.

NOTE 2—The selectivity of the column (separation of the nonaromatics from aromatics) can be increased by increasing the concentration of liquid phase or by increasing the temperature of the chromatographic column. The resolution of the aromatic compounds can be improved by increasing the length of the column or by decreasing the oven temperature or inlet pressure of the carrier gas.

5.1.2.3 Ethylbenzene must be separated from paraxylene and isopropylbenzene or from paraxylene plus isopropylbenzene with the depth of the valley after ethylbenzene not less than 50 % of the ethylbenzene peak height.

5.1.2.4 The system must measure durene with a peak height of at least 10 % of full scale chart deflection or at least 10 times the noise level.

NOTE 3—A combination of column materials and conditions that has been found to be particularly suitable for this test method is listed in Table 1. Such column may be obtained from most chromatography supply houses that offer packed GC columns.

5.2 Strip Chart Recorder Integrator—A recording potentiometer with a fullscale deflection of 10 mV or less should be used. If manual integration, such as triangulation, paper cut-out or planimeter, is employed the chart speed should be at least 1.5 m/h (60 in./h) in order to minimize errors in peak area measurement. This is not necessary where a ball-and-disk or an electronic integrator is employed.

5.3 Electronic Integration—Any electronic data processing system or integration device, or both, may be used to determine the areas of the chromatographic peaks.

TABLE 1 Typical Column and Conditions

Length, m	3.7 (12 ft)
Diameter, mm:	
Inside	...
Outside	3.2 (1/8 in.)
Liquid phase	CEF ^A
Weight % liquid	25
Solid support:	calcined pink, diatomaceous silica ^B
Mesh	80–100
Treatment	none
Inlet, °C	250
Detector, °C	250
Column, °C	110
Carrier gas	helium
Inlet pressure, KPa	550 (80 psi)
Flow rate, mL/min	35
Detector	flame ionization
Recorder, mV	1
Specimen, μ L	2
Sample split	(30 to 1)

^A *N,N*-bis (2-cyanoethyl) formamide.

^B The sole source of supply of the material Chromosorb-P known to the committee at this time is Celite Corp. 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.