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Standard Test Method for Composition of Turpentine by Gas Chromatography¹

This standard is issued under the fixed designation D 3009; 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 composition, specifically the α -pinene and β -pinene content, of wood, gum, and sulfate turpentine by packed column gas chromatography. Terms are defined in Terminology D 804.

1.2 *This standard does not purport to address all of the safety problems, 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.*

2. Referenced Documents

2.1 ASTM Standards:

D 13 Specification for Spirits of Turpentine²

D 804 Terminology Relating to Naval Stores, Including Tall Oil and Related Products²

3. Summary of Test Method

3.1 A weighted mixture of the sample and internal standard is prepared, an aliquot is injected into a 75°C chromatographic column, and the temperature programmed to 200°C to obtain the chromatogram. The peak areas for the components to be determined and also for the internal standard are measured. The percentages of the components present are calculated from the product of the peak area of the component, weight of internal standard, and the calibration factor divided by the product of the weight of sample and peak area of the internal standard.

4. Significance and Use

4.1 Earlier methods for characterizing turpentine were based on physical properties based on physical properties such as those in Specification D 13. As turpentine became widely used as a chemical raw material its chemical composition, especially its α -pinene and β -pinene content, became more important. The gas chromatographic technique described in this test method is a rapid and convenient method to perform this analysis. When measurement of the other minor component is required the use of capillary column gas chromatography is recommended.

5. Apparatus

5.1 *Linear Programmed Temperature Gas Chromatograph*—Any instrument with programming features and ei-

ther a thermal conductivity detector or a flame ionization detector and adaptable to the following conditions:

Detector cell temperature, °C	250
Injection port temperature, °C	200
Carrier gas (helium) flow at exit, cm ³ /min	50
Programmed temperature details:	
Starting column temperature, °C	75
	hold for 16 min
Column heating rate, °C/min	4
Upper column temperature, °C	200
	hold for 20 min

5.2 *Recorder*—A 1-mV recorder with a full-scale response of 1 s and a chart speed of 30 in./h.

5.3 *Integrating Equipment*—In order of preferences based on accuracy: electronic, disk, planimeter, or other acceptable methods.

5.4 *Column*—A 12-ft length of 3.18 to 6.35-mm ($\frac{1}{8}$ to $\frac{1}{4}$ -in.) outside diameter, 2.15 to 5.33-mm (0.085 to 0.210-in.) inside diameter, 0.51-mm (0.020-in.) wall, stainless steel tubing specially tempered for easy handling and packed with 20 weight % of polyethylene glycol having a molecular weight of 20 000³ on 60 to 80 mesh diatomaceous silica⁴ (nonacid washed). Condition at 210°C for about 4 h before use.

5.5 *Microsyringe*—Having a fixed needle, with a capacity large enough to deliver a sample sufficient for the chosen conditions.

6. Reagents

6.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵

6.2 Calibration Liquid:

6.2.1 α -pinene, purity of at least 99 %.

6.2.2 β -pinene, purity of at least 99 %.

6.3 *Column Packing*—Prepare a 20 weight % mixture of polyethylene glycol on 60/80 mesh diatomaceous silica in accordance with gas chromatographic practice.

6.4 *Liquid Phase*—polyethylene glycol.

6.5 *Solid Support*—diatomaceous silica (non-acid washed), 60/80 mesh.

6.6 *Reference Standard*—*n*-decane, purity of at least 99 %.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.34 on Naval Stores.

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² Annual Book of ASTM Standards, Vol 06.03.

³ Polyethylene glycol such as Carbowax 20M, manufactured by Union Carbide is available from most scientific supply houses.

⁴ Diatomaceous silica such as Chromosorb W, manufactured by Johns-Mansville Products Corp., is available from most scientific supply houses.

⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."