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Designation: D 3008 - 90

AMERICAN SOCIETY FOR TESTING AND MATERIALS

1916 Race St., Philadelphia, Pa. 19103
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Standard Test Method for Resin Acids in Rosin by Gas-Liquid Chromatography¹

This standard is issued under the fixed designation D 3008; 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 resin acids in tall oil, gum, or wood rosin after conversion to their methyl esters.

1.2 The composition is calculated from the ratio of each peak area to the total area of all peaks obtained. Rosins generally contain nonvolatile or polymeric materials for which no peaks are obtained under the conditions used. On the other hand, volatile neutral materials elute in the general region of resin acid methyl esters and can result in errors in the calculated composition. More reliable data is obtained by removal of neutral materials before gas chromatography.²

1.3 *This standard does not purport to address all of the safety problems 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. Specific hazards are given in Note 2.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water³

D 3257 Test Method for Aromatics in Mineral Spirits by Gas Chromatography³

E 260 Practice for Packed Column Chromatography⁴

3. Summary of Test Method

3.1 A portion of the sample is prepared by either converting it to the tetramethylammonium salts or esterifying it with diazomethane. A specimen of the prepared sample is injected into a 275°C injection port to a 200°C column of ethylene glycol succinate silicone copolymer (EGSS-X) or diethylene glycol succinate on silanized diatomaceous earth solid support. If added as the tetramethylammonium salt, the sample is pyrolyzed to the methyl ester. The pyrolyzed esters or the prepared esters are carried through the column with nitrogen or helium carrier gas and the chromatogram is obtained using a flame ionization detector.

3.2 The peak areas are obtained by any of the conventional means and the percent of the respective resin in acids is calculated from the ratios of the areas of the individual peaks to the total area of all peaks.

4. Apparatus

4.1 *Gas Chromatograph*—Any gas-liquid chromatograph having a flame ionization detector that can be operated at the conditions given in Table 1.

4.2 *Column*—Six to 10 ft (1.8 to 3 m) of 1/8-in. (3.2-mm) outside diameter, 0.085-in. (2.15-mm) inside diameter, 0.020-in. (0.52-mm) wall-thickness stainless steel tubing specifically tempered for easy handling and packed with the material as described in 8.1.

4.3 *Recorder*—A recording potentiometer with a full-scale deflection of 0 to 1-mV full-scale response time of 1 s or less, chart speeds of 1/2 to 1 in. (12 to 25 mm)/min, and sufficient sensitivity and stability to meet the requirements of 4.1.

4.4 *Specimen Introduction System*—Any system capable of introducing a representative specimen into the column. Microsyringes, 10- μ L capacity, with fixed needle have been found satisfactory.

4.5 *Integrating Equipment*—In order of preferences based on accuracy, electronic, disk, planimeter, or rule.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests unless otherwise specified. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

5.3 *Toluene*.

5.4 *N-Methyl-N-Nitroso-p-Toluenesulfonamide*.⁶

5.5 *Diethyl Ether, Anhydrous*.

5.6 *2-Ethoxyethanol*.

5.7 *Liquid Phase*—Diethylene glycol succinate (DEGS) or ethylene glycol succinate-silicone copolymer (EGSS-X, low-silicone content).

NOTE 1—EGSS-X is the preferred liquid phase because of its greater stability.

5.8 *Methanol*.

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

Current edition approved May 25, 1990. Published July 1990. Originally published as D 3008 - 72. Last previous edition D 3008 - 82.

² Zinkel, D. F., and Han, J., *Naval Stores Review*, Vol 96 (2), pp. 14-19 (1986).

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.01.

⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on 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."

⁶ Available from Aldrich Chemical Co., Inc., Milwaukee WI 53233 and Eastman Kodak, Rochester, NY 14650.