



Designation: D1240 – 24

Standard Test Methods for Rosin Acids Content of Pine Chemicals, Including Rosin, Tall Oil, and Related Products¹

This standard is issued under the fixed designation D1240; 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 These test methods cover the determination of rosin acids in tall oil, tall oil fatty acid, tall oil rosin, and other pine chemicals products.

1.2 These test methods may not be applicable to adducts or derivatives of rosin, fatty acid, or other pine chemicals products.

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

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

1.5 *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

D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D7795 Test Method for Acidity in Ethanol and Ethanol Blends by Titration

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

E177 Practice for Use of the Terms Precision and Bias in

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 D01.34 on Pine Chemicals and Hydrocarbon Resins.

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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.

ASTM Test Methods

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

3. Summary of Test Method

3.1 The rosin acids content is determined by one of two procedures; by selective esterification of fatty acids to form methyl esters followed by titration of the unreacted rosin acids, by selective esterification of fatty acids to form methyl esters followed by titration of the unreacted rosin acids.

4. Significance and Use

4.1 This is a revision of the method for measuring rosin acids content combines the three major ways of determining the rosin acids content of pine chemicals products into a single method.

4.1.1 For materials containing less than 15 % rosin, the modified Glidden procedure has gained acceptance. For materials containing more than 15 % rosin the modified Wolfe Method is preferred. The modified Wolfe and modified Glidden procedures differ only in their details. They have been combined here into a single procedure. This procedure can be run using either a potentiometer or an internal indicator to determine the end point of the titration. Use of a potentiometer is preferred and is the referee method. Use of an internal indicator is the principal alternative method. They will be referred to as the Potentiometric Method and the Internal Indicator Method.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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.³ Other grades may be used, provided it is first ascertained that the reagent is of

³ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Unless otherwise indicated, references to water shall be understood to mean deionized or distilled water.

6. Preparation of Sample

6.1 Homogeneous liquid materials may be used without further preparation.

6.2 Nonhomogeneous liquid materials should be heated until they are homogeneous, then a portion taken for analysis.

6.3 Solid samples are subject to surface oxidation which may affect the results. Prepare the sample for analysis by chipping small pieces from a freshly exposed surface of a lump or lumps and crush to a coarse powder to facilitate weighing and solution. Prepare fresh on the same day, prior to weighing, in order to avoid changes due to surface oxidation of crushed rosin on exposure to the air.

ROSIN ACIDS CONTENT BY THE POTENTIOMETRIC METHOD (Referee Method)

7. Scope

7.1 This test method covers the determination of rosin acids content of tall oil rosin, tall oil fatty acid, and other pine chemicals products, where the most reproducible results are desired. By using the potentiometric inflection end points, the error due to colorimetric end points is avoided.

8. Summary of Test Method

8.1 A sample is refluxed with methyl sulfuric acid to esterify the fatty acids. The rosin acids and sulfuric acid are then titrated potentiometrically, either with a use of pH meter or an automatic titrator and the rosin acids content calculated from the difference between the two inflection points obtained.

9. Apparatus

9.1 *pH Meter*—An indicating potentiometer having a limit of error not greater than ± 0.1 pH over a range from pH 1 to pH 13, using an alkali-resistant glass combination electrode (Note 1). The pH meter shall conform to the requirements of Test Method E70. Alternatively, an automatic potentiometric titrator may be used as described in Test Method D7795.

NOTE 1—A combination electrode will be used as the referee method and the saturated calomel half cell will be used as an alternative electrode for this method.

9.2 *Potentiometric Titrator*—Automatic titration systems capable of adding fixed increments of titrant at fixed time intervals (monotonic) or variable titrant increments with electrode stability between increment additions (dynamic) with endpoint seeking capabilities as prescribed in the method. At the very least, the automatic titration system shall meet the performance and specification requirements as warranted by the manufacturer, as described in Test Method D7795.

9.3 *Combination Electrodes*—Sensing electrodes may have the Ag/AgCl reference electrode built into the same electrode

body, which offers the convenience of working with and maintaining only one electrode. The combination electrode shall have a sleeve junction on the reference compartment and shall use an inert ethanol electrolyte, for example, 1M–3M LiCl in ethanol. These combination electrodes shall have the same response or better response than a dual electrode system. They shall have removable sleeves for easy rinsing and addition of electrolyte, as described in Test Method D664.

9.3.1 *Preparation of Electrodes*—When a Ag/AgCl reference electrode is used for the titration and it contains an electrolyte which is not 1M–3M LiCl in ethanol, replace the electrolyte. Drain the electrolyte from the electrode, wash away all the salt (if present) with water and then rinse with ethanol. Rinse several times with the LiCl electrolyte solution. Finally, replace the sleeve and fill the electrode with the LiCl electrolyte to the filling hole. When refitting the sleeve, ensure that there will be a free flow of electrolyte into the system. A combination electrode shall be prepared in the same manner. The electrolyte in a combination electrode can be removed with the aid of a vacuum suction.

9.3.2 *Testing of Electrodes*—Test the meter-electrode combination when first put into use, or when new electrodes are installed, and retest at intervals thereafter. Rinse the electrodes with solvent then with water, and dip them into a pH 4 aqueous buffer solution. Read the mV value after stirring 1 min. Remove the electrodes and rinse with water. Dip the electrodes into a pH 7 aqueous buffer. Read the mV value after stirring 1 min. Calculate the mV difference. A good electrode system will have a difference of at least 162 mV (20 °C to 25 °C). If the difference is less than 162 mV, lift the sleeve of the electrode and ensure electrolyte flow. Repeat the measurements. If the difference is still less than 162 mV, clean or replace the electrode(s).

9.3.3 When the sensing electrode and the reference electrode are separate, one pair of electrodes shall be considered as one unit. If one or the other is changed, it shall be considered as different pair and shall be retested.

9.3.4 *Maintenance and Storage of Electrodes*—Cleaning the electrodes thoroughly, keeping the ground-glass joint free of foreign materials, and regular testing of the electrodes are very important in obtaining repeatable potentials, since contamination may introduce uncertain erratic and unnoticeable liquid contact potentials. While this is of secondary importance when end points are chosen from inflection points in the titration curve, it may be quite serious when end points are chosen at arbitrarily fixed cell potentials.

9.3.5 Clean the glass electrode at frequent intervals based on use and type of samples being analyzed (not less than once every week during continual use) by immersing in nonchromium containing, strongly oxidizing cleaning solution. The reference electrode shall be cleaned periodically when in use or when a new electrode is installed. Drain the reference electrode at least once each week and refill with the fresh LiCl electrolyte as far as the filling hole. Ensure that there are no air bubbles in the electrode liquid. If air bubbles are observed, hold the electrode in a vertical position and gently tap it to release the