



Designation: D 1469 – 00

Standard Test Method for Total Rosin Acids Content of Coating Vehicles¹

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1. Scope

1.1 This test method covers the determination of the rosin acids content of rosin, unmodified by such materials as maleic or fumaric acid, or phenols. Rosin acids determined by this test method include free rosin.

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

1.3 *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.*

2. Summary of Test Method

2.1 The rosin acids content of tall oil rosin and related products is determined from a titration of the residual acidity of a specimen after first selectively esterifying any fatty acids, which may be present.

2.2 The specimen is refluxed in a solution of cyclohexane and butylsulfuric acid, with continuous removal of the water of esterification. The residual acidity, which consists of the rosin acids and the butyl sulfuric acid catalyst, is then titrated; a reagent blank is treated in the same manner and titrated to determine the amount of acid catalyst. The percent rosin acids is calculated as the difference between the two titrations².

2.3 This test method is applicable over the entire range of rosin acids-fatty acids concentrations.

3. Apparatus

3.1 *Air Condenser*, 760 mm (30 in.), with a 24/40 standard-taper joint.

3.2 *Buret*, self-zeroing, Class A, having a capacity of 25 mL, for the standard potassium hydroxide solution, fitted with soda-lime traps to protect against absorption of atmospheric carbon dioxide (CO₂).

3.3 *Erlenmeyer Flasks*, 250-mL capacity, with 24/40 ground joint.

3.4 *Moisture Collection Trap*, Bidwell-Sterling, 5-mL capacity, with 24/40 joints. Wrap with insulating tape.

3.5 *Dispensing Pipet*, 50-mL capacity, repeatability or reproducibility of $\leq 0.1\%$.

4. Reagents and Materials

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

4.2 *Cyclohexane*.

4.3 *n-Butyl Alcohol*.

4.4 *Butyl Alcohol-Sulfuric Acid Esterification Reagent*—Add 500 mL of *n*-butyl alcohol, 500 mL of cyclohexane, and 3.3 mL (6 g) of concentrated sulfuric acid (H₂SO₄) to a 2-L round-bottom flask with ground joint, connect to a moisture trap and condenser; then reflux on a hot plate for 30 min to distill out the water and to form butyl-sulfuric acid. Cool and store in a glass-stoppered bottle.

4.5 *Potassium Hydroxide, Alcoholic Standard Solution, 0.5 N (13.3 g/L)*—Dissolve 13.3 g of KOH pellets in 1 L of ethyl alcohol. Standardize against potassium acid phthalate primary standard.

4.6 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H₂SO₄).

4.7 *Thymol Blue Indicator Solution (10 g/L)*—Mix 1 g of thymol blue indicator with 100 mL of ethyl alcohol.

5. Procedure

5.1 Transfer to a 250-mL Erlenmeyer flask 1.0 to 1.2 g of sample, weighed to the nearest 0.0001 g.

5.2 Using a dispensing pipet, accurately measure 50 mL of the esterification reagent into the flask. Connect the flask to the moisture collection trap and condenser, place on a hot plate, heat to boiling, and reflux for 20 min AFTER the first drop of

¹ This test method is under the jurisdiction of ASTM Committee D01 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 Linder, A., and Persson, V., "Determination of Rosin Acids in Mixtures with Fatty Acids," *Journal, Am. Oil Chemists' Soc.*, Vol XXXIV, No. 1, 1957, pp. 24–27.

³ *Reagent Chemicals, American Chemical Society Specifications*, 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.