



Designation: D 1240 – 82

Standard Test Method for Rosin Acids in Fatty Acids¹

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1. Scope

1.1 This method covers the determination of rosin acids in fatty acids when the rosin acids content does not exceed 15 %.

2. Summary of Method

2.1 The rosin acids are determined by titration, after esterification of the fatty acids with methanol in the presence of sulfuric acid as a catalyst, followed by separation of the acids. The method requires use of an experimentally determined correction factor, dependent upon the amount of rosin acids present.²

3. Apparatus

3.1 *Flask*, 250-mL flat-bottom, of chemically resistant glass with a standard-taper 24/40 neck.

3.2 *Condenser*, water-cooled, with a joint fitting the flask described in 3.1.

3.3 *Separatory Funnel*, 500-mL, pear-shaped, fitted with a glass stopper.

3.4 *Buret*, 50-mL capacity, with 0.1-mL divisions.

4. Purity of Reagents and Water

4.1 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,³ where such specifications are available.

4.2 References to water shall be understood to mean distilled water.

5. Reagents

5.1 *Alcohol*—Neutral methanol, neutral isopropanol (91 to 99 %), neutral 95 % ethanol, or neutral denatured alcohol conforming to formula No. 30 or No. 3A of the U. S. Bureau of Internal Revenue.

5.2 *Ethyl Ether*.

5.3 *Methanol* (99.5 %).

5.4 *Methyl Orange Indicator Solution*—Dissolve 0.1 g of methyl orange in 100 mL of water.

5.5 *Phenolphthalein Indicator Solution*—Dissolve 1 g of phenolphthalein in 100 mL of methanol.

5.6 *Potassium Hydroxide Standard Alcoholic Solution* (0.2 *N* or 0.5 *N*)—Dissolve 13.3 g of KOH (preferably in pellet form) for a 0.2 *N* solution or 33.3 g for a 0.5 *N* solution (see 6.3) in methanol (99.5 %) and dilute to 1 L with methanol. Standardize to ± 0.001 *N*. The standardized solution should be protected against evaporation and the absorption of CO₂ from the air, and should be restandardized frequently.

5.7 *Sodium Sulfate Solution* (100 g Na₂SO₄/litre)—Dissolve 100 g of anhydrous Na₂SO₄ in water and dilute to 1 L. The resulting solution should have a pH of 7.2 ± 0.4 (Note 1).

NOTE 1—A suitable pH adjustment may be made by adding dilute (5 %) NaOH solution to a faint pink-red phenol phthalein color, then adding dilute H₂SO₄ (5 %) solution until the color is discharged.

5.8 *Sulfuric Acid* (*sp gr* 1.84).

6. Procedure

6.1 Dissolve 40 ± 0.1 g of the sample in 100 mL of methanol in a 250-mL flat-bottom flask. Twirl the flask to dissolve the oil and add a clean boiling chip. Add slowly 5 mL of H₂SO₄, while swirling the flask vigorously. Connect the flask to the condenser, apply heat, and reflux the contents for 10 min. Cool the flask to room temperature with cold water.

6.2 Add 250 mL of the Na₂SO₄ solution to a 500-mL separatory funnel. Pour the contents of the flask into the funnel and complete the quantitative transfer of the flask contents with 100 mL of ether. Thoroughly shake the mixture in the funnel. Allow to settle, draw off the salt layer, and discard. Wash the contents of the funnel twice again with 250-mL portions of Na₂SO₄ solution. The last washing should not react pink to methyl orange indicator.

6.3 After removing the last wash, drain the contents of the separatory funnel into a 500-mL Erlenmeyer flask. Rinse the funnel with 20 mL of ether and add the rinsings to the flask. Add 20 mL of ethyl alcohol and 1 mL of the phenolphthalein indicator solution. Titrate to the appearance of a pink-red color using 0.2 *N* alcoholic KOH solution if the rosin acids content is less than 5 % or 0.5 *N* if more than 5 %.

¹ This 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.

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² For information on the development of this method and the establishment of the correction factor, see Herrlinger, R. and Compeau, G. M., "The Determination of Rosin in Fatty Acids," *Journal of the American Oil Chemists' Society*, JAOCA, August 1952, pp. 342–344.

³ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, D. C. 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, N. Y., and the "United States Pharmacopeia."

7. Calculation

7.1 Calculate the percentage of rosin acids as follows:

$$\text{Rosin acids, \%} = (1.031 \times AN \times 30.2)/S - 0.74$$

where:

- A = millilitres of KOH solution used for titration of the sample,
- N = normality of KOH solution,
- S = grams of sample used, and
- 0.74 = correction factor² for a rosin acids content not over 15 %.

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