



Standard Test Method for Total Rosin Acids Content of Coating Vehicles¹

This standard is issued under the fixed designation D 1469; 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 total rosin acids content of rosin esters, varnishes, and alkyd resins, unmodified by such materials as maleic or fumaric acid, or phenols. Total rosin acids determined include free rosin, esterified rosin, and metallic salts of rosin.

1.2 This test method is primarily designed for material containing 0.5 to 5 % rosin on the nonvolatile basis.

1.3 *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 1193 Specification for Reagent Water²

D 1259 Test Methods for Nonvolatile Content of Resin Solutions³

D 1644 Test Methods for Nonvolatile Content of Varnishes³

3. Summary of Test Method

3.1 The specimen is saponified with potassium hydroxide-ethylene glycol reagent and acidified with hydrochloric acid. Heat is applied to hydrolyze metallic driers. This is necessary, especially when metallic rosinate are present.

3.2 The mixture described in 3.1 is extracted with benzene (Note 1). The rosin and fatty acids and unsaponifiables pass into the benzene layer. The aqueous layer will contain certain dibasic acids, polyhydric alcohols, and other water-soluble products of saponification.

NOTE 1—While it has not been evaluated for this procedure, toluene has been found to be an acceptable alternative in similar procedures.

3.3 The benzene is removed by evaporation, the residue weighed, and the rosin acids are determined by a selective esterification and titration method.⁴

4. Apparatus

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

4.2 *Buret*, automatic type, 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).

4.3 *Erlenmeyer Flasks*, 250 and 500-mL capacity, with 24/40 ground joint.

4.4 *Separatory Funnels*, three of 1-L capacity.

4.5 *Steam Bath*, located in a fume hood, for evaporation of volatile solvents.

4.6 *Moisture Collection Trap*, constructed according to details shown in Fig. 1. Wrap with ½-in. (12.7-mm) asbestos tape.

4.7 *Pipet*, automatic, 50-mL capacity.⁵

5. Reagents and Materials

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.⁶ 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 as defined by Type II of Specification D 1193.

5.3 *Benzene*—(Toluene may be an acceptable substitute, see Note 1.)

5.4 *Butyl Alcohol-Sulfuric Acid Esterification Reagent*—Add 500 mL of *n*-butyl alcohol, 500 mL of benzene, and 3.3 mL (6 g) of sulfuric acid (H₂SO₄) to a 2-L round-bottom flask with ground joint, connect to a moisture trap and condenser, and reflux on a hot plate for 30 min to distill out the water and

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 D01.33 on Polymers and Resins.

Current edition approved April 15, 1993. Published June 1993. Originally published as D1469 – 57 T. Last previous edition D1469 – 73 (1988)^{ε1}.

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ 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.

⁵ Pipets obtainable from the Arthur H. Thomas Co., W. Washington Square, Philadelphia, PA 19105 Catalog No. 8212, or from the Scientific Glass Apparatus Co., Catalog No. JP-6000, have been found satisfactory for this purpose.

⁶ *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.