



Designation: D 1397 – 93 (Reapproved 1998)

Standard Test Method for Unsaponifiable Matter in Alkyd Resins and Resin Solutions¹

This standard is issued under the fixed designation D 1397; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of unsaponifiable matter in alkyd resins and resin solutions. This test method is not applicable to alkyd resins containing modifying agents such as urea, melamine, phenols, rosin, and styrene.

1.2 *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.* For a specific hazard statement, see Note 4.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

3. Significance and Use

3.1 The unsaponifiable matter in alkyd resins controls the properties of the final film.

4. Apparatus

4.1 *Aluminum Beaker*, having a capacity of 125 mL.³

4.2 *Flask and Condenser*—A 200-mL Erlenmeyer flask fitted with a water-cooled glass reflux condenser. The connection between the flask and condenser shall be a standard 24/40 taper ground-glass joint.

4.3 *Separatory Funnels*—Three 500-mL capacity fitted with standard-taper, ground-glass stoppers and stopcocks. Stopcocks should be lubricated sparingly with ether-insoluble stopcock grease. Alternatively, funnels fitted with tetrafluoroethylene (TFE-fluorocarbon) stopcocks may be used.

4.4 *Steam Bath.*

4.5 *Vacuum Drying Oven*—A small, laboratory-size vacuum oven, thermostatically controlled to operate at $80 \pm 5^\circ\text{C}$. A water aspirator vacuum source is satisfactory.

5. Reagents and Materials

5.1 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.⁴ 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 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.*

5.4 *Benzene-Alcohol Mixture*—Mix equal volumes of benzene and ethyl alcohol, add 2 drops of phenolphthalein indicator solution, and neutralize with 0.02 N sodium hydroxide (NaOH) solution to a persistent faint pink color.

5.5 *Ethyl Alcohol (95 volume %)*—Pure ethyl alcohol or denatured alcohol conforming to Formula No. 2B of the U. S. Bureau of Internal Revenue.

5.6 *Ethyl Ether.*

5.7 *Phenolphthalein Indicator Solution (10 g/L)*—Dissolve 1 g of phenolphthalein in ethyl alcohol (95 %) and dilute to 100 mL with ethyl alcohol.

5.8 *pH Indicator Paper*, universal type.

5.9 *Sodium Hydroxide Solution (50 %)*—Dissolve sodium hydroxide (NaOH) in an equal weight of water.

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.

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

³ Aluminum beakers, Catalog No. 2100, manufactured by the A. H. Thomas Co., W. Washington Square, Philadelphia, PA 19105, have been found satisfactory for this purpose.

⁴ *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.