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Standard Test Method for Isophthalic Acid in Alkyd and Polyester Resins¹

This standard is issued under the fixed designation D 2690; 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 gravimetric determination of the isophthalic acid content of alkyd resins and polyesters. There is no interference from styrene monomer or polymer or from other dicarboxylic acids except terephthalic acid.
- 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 Section 7.

2. Referenced Documents

- 2.1 ASTM Standards:
- D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 The resin is saponified with alcoholic potassium hydroxide and benzene to precipitate entirely the potassium salt of the phthalic acid isomer. Since the salt contains entrained impurities, it is dissolved in water and diluted to volume. An aliquot portion is acidified under conditions that release the insoluble acid in filterable form. It is then isolated, weighed, and corrected for its slight solubility in water.

4. Significance and Use

4.1 This test method is used to determine the amount of isophthalic acid contained in alkyd and polyester resins. Use of this test method provides a means whereby the relative applicability of the alkyd or polyester resin to the particular end use may be estimated by the buyer and the seller.

5. Apparatus

- 5.1 Flask and Condenser—A 250-mL Erlenmeyer flask fitted with an air-cooled glass reflux condenser 30 in. (760 mm) in length. The connection between the flask and condenser should be a standard-taper 24/40 ground joint.
 - 5.2 Heat Source:
 - 5.2.1 Combination Hot Plate and Magnetic Stirrer, or
- 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.

- 5.2.2 Heating Mantle and Magnetic Stirrer Base and Bar.
- 5.3 Büchner Funnel, fritted glass, medium porosity, 150-mL capacity.
- 5.4 Volumetric Flask, 100-mL volume.
- 5.5 Fritted-Glass Filter Crucible, medium porosity, 30-mL capacity.

6. Reagents

- 6.1 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.
- 6.2 *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.
- 6.3 Alcohol-Benzene Wash Solution (2 + 3)—Mix absolute ethyl alcohol (Note 1) with benzene in the proportion of 2 volumes of alcohol to 3 volumes of benzene.

Note 1—The alcohol may be denatured Formula 2-B, but must be anhydrous.

- 6.4 Alcoholic Potassium Hydroxide Solution (0.5 N)—Dissolve 33 g of KOH in 1 L of absolute ethyl alcohol (Note 1) by reflux or by standing overnight. Protect against carbon dioxide absorption. Filter just before use.
 - 6.5 Benzene (anhydrous).
 - 6.6 Ether (anhydrous).
- 6.7 Hydrochloric Acid (sp gr 1.19)—Concentrated hydrochloric acid (HCl).
 - 6.8 Methyl Purple Indicator Solution.

7. Hazards

7.1 The reagents and samples used in this test method may, under some conditions, be hazardous. Refer to the manufacturer's material safety data sheets for specific handling and safety precautions. Safe laboratory handling procedures and all

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.