



Designation: D 1306 – 88 (Reapproved 1996)^{ε1}

Standard Test Method for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Gravimetric)¹

This standard is issued under the fixed designation D 1306; 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.

^{ε1} NOTE—Keywords were added editorially in October 1996.

1. Scope

1.1 This test method covers the gravimetric determination of phthalic anhydride in alkyd resins and esters that contain dibasic acids such as maleic, fumaric, adipic, and sebacic, which would interfere if Test Method D 563 was used.

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.*

2. Referenced Documents

2.1 ASTM Standards:

- D 563 Test Method for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions²
- D 1193 Specification for Reagent Water³

3. Summary of Test Method

3.1 The specimen is saponified with alcoholic potassium hydroxide and benzene to precipitate quantitatively the potassium salt of phthalic acid as $C_6H_4(COOH)_2 \cdot C_2H_5OH$. Interfering substances are eliminated by dissolving the precipitate in water, adjusting the pH of the solution to 2.5 with nitric acid, and filtering. Phthalic acid is then precipitated as nonstoichiometric lead phthalate and calculated to phthalic anhydride, using a factor obtained when compositions of known purity were analyzed similarly.

4. Significance and Use

4.1 The phthalic anhydride content of alkyd resins controls the properties of the final film.

5. Apparatus

5.1 *Flask and Condenser*—A 500-mL Erlenmeyer flask fitted with an air-cooled glass reflux condenser 30 in. (760 mm) in length. The connection between the flask and condenser shall be a standard-taper 24/40 ground-glass joint.

5.2 *Water Bath.*

5.3 *Guard Tube*, filled with soda lime.

5.4 *Fritted-Glass Filter Crucible*, medium porosity, of 30-mL capacity.

5.5 *Filter Flasks*, suction-type.

5.6 *Crucible Holder.*

5.7 *Oven*, of gravity convection type.

5.8 *Desiccator*, containing concentrated H_2SO_4 (sp gr 1.84) as the desiccant.

5.9 *Flash Filtrator.*

5.10 *pH Test Assembly.*

5.11 *Volumetric Flask*, 100-mL.

5.12 *Erlenmeyer Flask*, 250-mL, wide-mouth, with glass stopper not smaller than a No. 27.

5.13 *Delivery Pipet*, 2-mL.

6. Reagents

6.1 *Purity of Reagent*—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.

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 06.03.

³ *Annual Book of ASTM Standards*, Vol 11.01.

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 Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.