Designation: D 563 – 88 (Reapproved 1996)ε1

An American National Standard

Standard Test Method for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions[1]

This standard is issued under the fixed designation D 563; 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.

ε[1] NOTE—Keywords were added editorially in October 1996.

1. Scope

1.1 This test method covers the determination of the phthalic anhydride content (Note 1) of alkyd resins and resin solutions, including those containing styrene.

NOTE 1—This test method is not applicable for the determination of phthalic anhydride in alkyd resins containing other dibasic acids such as maleic or fumaric, or modifying agents such as urea, melamine, and phenolic resins.

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 1193 Specification for Reagent Water[2]

3. Significance and Use

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

4. Apparatus

4.1 *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 24/40 standard taper ground-glass joint.

4.2 *Water Bath*.

4.3 *Fritted-Glass Filter Crucible*, fine or medium porosity, of 30-mL capacity.

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

4.5 *Filter Flasks*.

4.6 *Crucible Holder*.

5. Reagents

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.[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.

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 *Alcohol-Benzene Wash Solution*—Mix one volume of absolute ethyl alcohol (Note 2) with three volumes of benzene.

NOTE 2—The alcohol may be denatured, formula 2B, but must be absolute.

5.4 *Benzene*.

5.5 *Ether*—Anhydrous analytical reagent-grade ether.

5.6 *Hydrochloric Acid, Standard* (0.1 *N*).

5.7 *Potassium Hydroxide, Alcoholic Solution*—Dissolve 66 g of reagent-grade potassium hydroxide (KOH) in 1 L of absolute ethyl alcohol (Note 2). Filter just before use.

6. Procedure

6.1 Weigh by difference, from a closed container into the 500-mL Erlenmeyer flask, a specimen of resin or resin solution sufficient to yield from 0.8 to 1.2 g of potassium alcohol phthalate. Add 150 mL of benzene, warming slightly on the steam bath if necessary, to effect solution. Add 60 mL of alcoholic KOH solution and attach the condenser. Place the flask in a water bath to a depth approximately equal to that of

[1] This 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] *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.