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An American National Standard

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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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 the contents of the flask. Warm the bath, maintaining a temperature of 40°C for 1 h, then gradually raise the temperature until the alcoholic solution boils gently. Reflux for 1½ h.

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[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.

6.2 Remove the flask from the bath and wash down the inside of the condenser with a few millilitres of alcohol-benzene wash solution. Remove the condenser, cap the flask with the soda-lime guard tube, and cool by means of running water or an ice bath.

6.3 When cool, filter immediately and as rapidly as possible, through a fritted-glass crucible that previously has been tared, using the alcohol-benzene wash solution for transferring the precipitate and washing the reaction flask. Wash the precipitate with successive portions of alcohol-benzene wash solution until a few millilitres of washings collected in a second suction flask are no longer alkaline to phenolphthalein. (Normally about 75 mL of wash solution are sufficient.) Do not allow air to be drawn through the crystals, as they are hygroscopic. Finally pour 25-mL of ether into the crucible and draw through the precipitate with the aid of suction.

6.4 Wipe the outer surface of the crucible with a clean cloth and place in a gravity convection oven at 60°C for 1 h (Note 3). Cool to room temperature in a desiccator and weigh.

NOTE 3—The precipitate is the alcoholate ($C_6H_4(COOH)_2 \cdot (C_2H_5OH)$), and the alcohol of crystallization will be slowly driven off on prolonged heating. It is safe, however, to dry the alcoholate at temperatures up to 60°C for as long as 1 h.

6.5 *Correction for Carbonates*—Coprecipitation of potassium carbonate (K_2CO_3) with the potassium alcohol phthalate may be a source of error. If a correction for K_2CO_3 is desired, proceed as follows: Dissolve the weighed precipitate in about 50 mL of distilled water that has been neutralized to phenolphthalein. Add 3 to 4 drops of phenolphthalein indicator solution, and if the solution is alkaline, titrate with 0.1 *N* HCl.

7. Calculation

7.1 Calculate the percent of phthalic anhydride *A* in the specimen as follows:

$$K = VN \times 0.1382$$

$$A = [((P - K) \times 0.5136)/S] \times 100$$

where:

K = correction for K_2CO_3 (if determined), g,
V = HCl used for titration (see 6.5), mL,
N = normality of HCl,
P = potassium alcohol phthalate (see 6.5), g, and
S = specimen used, g.

8. Precision and Bias

8.1 Precision and bias are being determined.

9. Keywords

9.1 alkyd resins; phthalic anhydride content; resin solutions