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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Isophthalic Acid in Alkyd and Polyester Resins[1]

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 Water[2]

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

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

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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.

applicable OSHA regulations are to be followed.

8. Procedure

8.1 Weigh a portion of resin containing about 400 mg of isophthalic acid (usually 1 to 3 g of vehicle) into a 250-mL Erlenmeyer flask and dissolve in 10 mL of benzene. Add 100 mL of freshly filtered 0.5 *N* alcoholic KOH solution and insert a magnet stirrer bar. Attach the air condenser and reflux for 1½ h with constant stirring by means of a heating mantle or combination stirrer-hot plate.

8.2 Remove from the source of heat, remove the condenser, and fill the flask to the neck with benzene. Cool the solution to room temperature with stirring, allow to settle a few minutes, then filter with suction through a 150-mL fritted glass Büchner funnel of medium porosity. Transfer and wash to bulk of the precipitate with alcohol (2 + 3). Rinse the flask with about 25 mL of ethyl ether and add to the funnel, draining thoroughly with suction.

8.3 Dry the funnel and flask in a 105°C oven for about 30 min. Dissolve the dipotassium salts in small portions of water by first adding them to the dried flask to remove all traces of the precipitated salts, and then to the funnel collecting them in a clean suction flask. Transfer the filtrate with water to a 100 mL volumetric flask and dilute to the mark.

8.4 Mix the solution thoroughly and withdraw a 50-mL aliquot by means of a pipet and transfer to a 125-mL Erlenmeyer flask. If it is necessary to use smaller aliquots, make the volume up to 50 mL with water. Add a magnetic stirrer bar and about 2 drops of methyl purple indicator solution and while stirring acidify the solution by adding HCl (sp gr 1.19) dropwise until the color changes, then add 7 drops in excess.

8.5 Continue the stirring for about 5 min and collect the precipitated isophthalic acid in a weighed fritted-glass filter crucible of medium porosity. Do not wash or add any water at this time; instead, collect the filtrate in a clean flask and use it in five 10-mL portions to transfer the precipitate quantitatively to the crucible. The filtrate may be reused if necessary. Now wash the collected solution in the crucible twice with 3-mL portions of distilled water added from a pipet and directed against the sides of the crucible while rotating so as to effect maximum washing with the two small measured volumes of water. Dry the crucible in an oven at 105°C for 1 h, cool in a desiccator, and weigh. If the collected isophthalic sample exceeds 200 mg, withdraw a smaller aliquot from the remaining solution, dilute to 50 mL with water and repeat the precipitation procedure. If less than 50 mg are collected, repeat from the beginning with a larger resin specimen.

9. Calculation

9.1 Calculate the percent of isophthalic acid *I* as follows:

$$I = \frac{(P + 0.005) \times 100 \times 100}{SWA} \quad (1)$$

where:

P = weight of precipitate, g,
S = weight of resin specimen, g,
W = nonvolatile content expressed as a decimal, and
A = size of aliquot, mL (usually 50).

10. Precision and Bias

10.1 *Precision*—On the basis of an interlaboratory study in which one operator in each of six laboratories made duplicate determinations at each of six laboratories on two samples of different manufacture, the within and between laboratory standard deviations were found to be:

	Standard Deviation, %	
Isophthalic Acid Content, %	Within Laboratory	Between Laboratory
30	0.025	0.18
50	0.083	0.44

Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

10.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than the following:

Isophthalic Acid Content, %	Differences, %
30	0.15
50	0.50

10.1.2 *Reproducibility*—Two results, each the mean of duplicate measurements, obtained by operators in different laboratories should be suspect if they differ by more than the following:

Isophthalic Acid Content, %	Differences, %
30	0.6
50	1.5

10.2 *Bias*—Bias canot be determined as no reference material is available.

11. Keywords

11.1 alkyd resins; gravimetric; isophthalic acid; polyester resins