Designation: D 2689 – 88 (Reapproved 1999)

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practices for Testing Alkyd Resins[1]

This standard is issued under the fixed designation D 2689; 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 These practices cover test methods for testing alkyd resins as listed in Table 1. They are the test methods most often used to characterize alkyds. All of the analytical test methods were subjected to interlaboratory testing using oil-modified alkyds. Their adaptability to other types of alkyds has not been studied. Although each test method specifies a recommended amount of specimen for starting a separate analysis, several of the procedures can be conducted on the same starting specimen, if desired. For example, the tests for unsaponifiable matter, fatty acid content, oil identification, and phthalic acid content could all be run on a consecutive basis, if all were required.

2. Referenced Documents

2.1 *ASTM Standards:*

D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester[2]

D 563 Test Method for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions[3]

D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)[4]

D 1259 Test Methods for Nonvolatile Content of Resin Solutions[5]

D 1306 Test Method for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Gravimetric)[3]

D 1397 Test Method for Unsaponifiable Matter in Alkyd Resins and Resin Solutions[3]

D 1398 Test Method for Fatty Acid Content of Alkyd Resins and Alkyd Resin Solutions[3]

D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products[5]

D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)[5]

D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method[3]

TABLE 1 Test Methods for Testing Alkyd Resins

Test Method	Section	ASTM Designation
Nonvolatile content	4	D 1259
Acid value	5	D 1639
Unsaponifiable matter	6	D 1397
Fatty acid content	7	D 1398
Identification of oils and acids	8	D 2245
Phthalic anhydride content	9	D 563
		D 1306
Isophthalic acid content	10	D 2690
Polyhydric alcohol content	11	D 2998
Identification of carboxylic acids	12	D 2455
Flash point	13	D 93
		D 3278
Color	14	D 1209
		D 1544
Density	15	D 1475
Viscosity	16	D 1545
Clarity	17	D 2090

D 1639 Test Method for Acid Value of Organic Coating Materials[3]

D 2090 Test Method for Clarity and Cleanness of Paint and Ink Liquids[3]

D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints[5]

D 2455 Test Method for Identification of Carboxylic Acids in Alkyd Resins[3]

D 2690 Test Method for Isophthalic Acid in Alkyd and Polyester Resins[3]

D 2998 Test Method for Polyhydric Alcohols in Alkyd Resins[3]

D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus[5]

3. Significance and Use

3.1 These practices should be used as a reference for any alkyd resin analyst who wants general information about classifying alkyd resins. In each case, the significance and use of the specific classification item will be found in the referenced test method.[6]

4. Nonvolatile Content

4.1 A unique test method for determining nonvolatile matter in solutions of alkyd resins in volatile organic solvents provides for the drying of very thin films of resin quickly, thus

[1] These practices are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved May 27, 1988. Published October 1988. Originally published as D 2689 – 68. Last previous edition D 2689 – 80.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 06.03.

[4] *Annual Book of ASTM Standards*, Vol 06.04.

[5] *Annual Book of ASTM Standards*, Vol 06.01.

[6] *Annual Book of ASTM Standards*, Vols 06.02 and 06.03.

minimizing chances for volatiles to be trapped or drying oils to oxidize. Test Method A of Test Methods D 1259, was thoroughly tested with alkyd resins with outstanding repeatability of 0.1 % and reproducibility of 0.2 %. This procedure, sometimes referred to as the "foil" test method, should be considered the "referee" test method for all alkyd analyses requiring maximum precision.

5. Acid Value

5.1 Acid value of an alkyd resin is expressed as the number of milligrams of potassium hydroxide required to neutralize the free acidity of 1 g of nonvolatile material under the conditions of the test. Test Method D 1639, was especially prepared for this purpose.

6. Unsaponifiable Matter

6.1 The unsaponifiable matter in alkyd resins is the ethyl ether-soluble, water-insoluble portion that remains after a relatively large specimen has been subjected to an aqueous-alcoholic saponification. The yield is normally low and is an indication of purity insofar as the presence of modifying resins will affect the result. Certain other resins can interfere due to variations in degree of saponification and solubility in ether. The test method is therefore not applicable to alkyds containing such other resins as styrene, rosin, phenols, and formaldehyde condensates. The unsaponifiable matter is determined gravimetrically in accordance with Test Method D 1397 and may be calculated on the nonvolatile or solution basis according to prior agreement.

7. Fatty Acid Content

7.1 The total fatty acids in alkyd resins can be measured gravimetrically on a separate specimen in accordance with Test Method D 1398. This test method provides for the isolation of fatty acids after saponification under anhydrous conditions, removal of the salts of the dicarboxylic acid by filtration, removal of the unsaponifiable matter and original solvents by benzene extraction of the filtrate, and finally ether extraction of the acidified aqueous layer that remains. The test method was prepared for use with orthophthalic alkyds and, if isophthalic acid is present, a considerable amount will appear in the extracted oil acids where it is readily detected as white crystals. Interference from isophthalic acid can be avoided by redissolving the oil acids in benzene, filtering into a weighed beaker and drying as before. Some modifying agents such as urea, melamine, phenols, rosin, and styrene will contaminate the isolated fatty acids in varying degrees and Test Method D 1398 is considered inapplicable in the presence of such other resins. If the fatty acids isolated by Test Method D 1398 are to be examined further for identification (Section 8), a small, weighed crystal of hydroquinone to serve as an antioxidant, should be added prior to the evaporation of the ether.

8. Identification of Oils and Oil Acids

8.1 The oils and oil acids contained in oil-modified alkyd resins can be identified by gas chromatographic separation of their methyl esters that are formed in the presence of margaric acid added as an internal standard. The test method is not applicable to fatty acids that have polymerized or oxidized to such an extent that no characteristic monomeric fatty acids remain. The test is conducted in accordance with Method D 2245, and the sample may be obtained as described therein or from material remaining from the fatty acid determination of Section 7.

9. Phthalic Anhydride Content

9.1 Alkyd resins that are known to contain phthalic anhydride as the only dicarboxylic acid and are not modified with resins such as phenolics, urea- or melamine-formaldehyde may be analyzed in accordance with Test Method D 563. Styrene does not interfere so that styrenated alkyds may be tested by the same test method.

9.2 Determination of phthalic anhydride in alkyd resins that contain isomers of phthalic acid or other dibasic acids such as maleic, fumaric, etc., must be made in accordance with Test Method D 1306, in which the phthalic anhydride is determined gravimetrically as the lead salt.

10. Isophthalic Acid Content

10.1 A rapid and simple procedure for the gravimetric determination of isophthalic acid in alkyd resins is provided in Test Method D 2690. There is no interference in this test method from styrene monomer of polymer or from other dicarboxylic acids except terephthalic acid, which is recovered quantitatively.

11. Polyhydric Alcohol Content

11.1 The polyhydric alcohols in alkyds can be determined qualitatively and quantitatively in accordance with the procedure in Test Method D 2998. The resin to be analyzed is dried, subjected to aminolysis, and treated to form trimethylsilyl ether derivatives of the alcohols which are separated by gas-liquid chromatography. An internal standard is used for quantitative purposes. Flame-ionization detectors cannot be used with Test Method D 2998.

12. Identification of Carboxylic Acids

12.1 Method D 2455 describes a procedure for the qualitative determination of all the carboxylic acids in alkyd resins, including resin-modified alkyds. This procedure makes use of gas chromatographic separation of the methyl esters of the respective acids that are formed by direct transesterification of the resin with lithium methoxide. The acids, including the fatty acids, are identified by their relative retention time which is given for 23 acids and others may be added if desired. Since maleic and fumaric acids react differently than the other acids encountered, an alternative procedure is supplied for their differentiation. It is sometimes possible to identify the drying oils in the alkyd from the fatty acid esters that appear in this procedure, but it is not as reliable for oil identification as Method D 2245 (Section 8).

13. Flash Point

13.1 In the Setaflash closed tester, a small specimen is injected by means of a syringe and its flash temperature is determined visually, as described in Test Method B of Test Method D 3278. This procedure is faster, more convenient, and somewhat more precise than the Pensky-Martens procedure

described in Test Method B of Test Method D 93; however, results obtained by either test method are acceptable.

14. Color

14.1 The Gardner color method, Test Method D 1544, compares a specimen to glass filters by means of a comparator, while Test Method D 1209 compares the specimen to calibrated liquids in Nessler tubes. It is recommended that Test Method D 1544 (Gardner Color Scales) be used for the color specification where practical. Where particularly light color is required, Test Method D 1209 (platinum-cobalt scale), may be used when agreed upon between the producer and the user. There is no reliable correlation between the two test methods. In general, Test Method D 1209 is to be preferred for light-colored liquids while Test Method D 1544 is generally used for darker liquids, such as drying oils, varnishes, and coating resins.

15. Density

15.1 Test Method D 1475 describes the determination of density by either the pycnometer or weight cup test methods. Either test method is acceptable for alkyd resins.

16. Viscosity

16.1 The viscosity of resin solutions may be determined by the bubble-time test method, in accordance with Test Method D 1545. Viscosity is measured at 77°F (25°C) by comparing the time for a bubble to traverse a standard tube containing the resin, to the time for standard oils under identical conditions. The bubble seconds are approximately equal to stokes and may be converted to poises by dividing by density.

17. Clarity

17.1 Clarity may be determined in accordance with Test Method D 2090, wherein a specimen is placed in a bubble viscosity tube and rated subjectively for seven factors relating to clarity.

18. Keywords

18.1 acid value; alkyd resin; clarity; fatty acid; isophthalic acid; oil acids; oils; phthalic anhydride; polyhydric alcohol; unsaponifiable matter