Designation: D 1398 – 93 (Reapproved 1998)

Standard Test Method for Fatty Acid Content of Alkyd Resins and Alkyd Resin Solutions[1]

This standard is issued under the fixed designation D 1398; 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.

1. Scope

1.1 This test method covers the gravimetric determination of the total fatty acids in alkyd resins and alkyd resin solutions. The test method is not applicable to alkyd resins containing modifying agents such as urea, melamine, phenols, rosin, and styrene.

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 whoever uses this standard to consult and 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 Solutions[2]

D 1193 Specification for Reagent Water[3]

D 1615 Test Methods for Glycerol, Ethylene Glycol, and Pentaerythritol in Alkyd Resins[2]

D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints[4]

3. Significance

3.1 This test method is used to determine total fatty acid content of alkyl resins and this solution, in the absence of interfering compounds, as a means whereby the relative applicability of the alkyd resin to the particular end use may be estimated by buyer and seller.

4. Apparatus

4.1 *Beakers*, having capacities of 150 and 400 mL.

4.2 *Desiccator*,

4.3 *Filter Paper*, rapid, low-ash filter paper, to fit the filtering funnel.

4.4 *Filtering Funnel*, 75-mm diameter, long-stem.

4.5 *Flask*, 250-mL, flat-bottom, with standard taper opening.

4.6 *Nitrogen Gas Supply*.

4.7 *Separatory Funnels*, three 500-mL capacity glass-stoppered, fitted with standard-taper ground-glass stoppers and stopcocks.

4.8 *Steam Bath*.

4.9 *Water Bath*.

4.10 *Vacuum Drying Oven*, small, laboratory-size, thermostatically controlled to operate at 60± 2°C. A water-aspirator vacuum source is satisfactory.

4.11 *Cotton*, absorbent, USP.

5. Reagents and Materials

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.[5] 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 conforming to Type IV of Specification D 1193.

5.3 *Ether*, anhydrous.

5.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.5 *Indicator Paper*, universal-type.

5.6 *Sodium Sulfate* (Na_2SO_4), anhydrous.

6. Procedure

6.1 Saponify a portion of resin or resin solution in accordance with Test Method D 563. After filtering the potassium

[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.

Current edition approved April 15, 1993. Published June 1993. Originally published as D 1398 – 56. Last previous edition D 1398 – 84.

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

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

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

[5] *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.

phthalate alcoholate (6.3), transfer the combined filtrate and washings to the 400-mL beaker with the aid of 25 mL of water from a wash bottle. Concentrate to a volume of approximately 25 mL on the steam bath under a blanket of nitrogen to prevent oxidation of the fatty acids. (**Caution**—Be sure to use a hood.) Transfer to a 500-mL separatory funnel with the aid of water from a wash bottle, dilute with water to approximately 300 mL, and add 10 mL of alcohol.

6.2 Extract the unsaponifiable and volatile thinners with successive 50-mL portions of ether (not less than three, or until a colorless ether extract is obtained), combining the ether extracts in the first separatory funnel and using the other two funnels for the successive extractions (Note 1). Finally, wash the combined ether extracts with three 15-mL portions of water, adding the water washes to the main aqueous phase. Discard the combined ether extracts.

NOTE 1—If the layers do not separate easily, carefully draw off the lower, clear aqueous layer and add 2 to 3 mL of alcohol, by means of a pipet, to the ether emulsion phases in the separatory funnel. Swirl gently to break the emulsion and continue to draw off the lower layer. This procedure for breaking the emulsion may be repeated on subsequent extractions, if necessary.

6.3 Acidify the aqueous phase to a pH of approximately 2 by slowly adding HCl, cooling under running tap water. When the mixture has cooled to room temperature, extract the fatty acids with successive 25-mL portions (not less than three) of ether until a colorless ether extract is obtained, combining the ether extracts in the first separatory funnel and using the other two funnels for the successive extractions. Wash the combined ether extracts with successive 10-mL portions of water until free of mineral acid when tested with an indicator paper. Discard the aqueous phase (unless Test Method D 1615 is being followed).

6.4 Dry the combined ether extracts in the separatory funnel by the addition of successive small quantities of anhydrous Na_2SO_4.

NOTE 2—The free water has been removed when, by the addition of a small quantity of Na_2SO_4 and gentle swirling, the excess Na_2SO_4 is seen to disperse as a freely moving powder.

6.5 By either of the following methods, filter the ether extract portionwise into a tared (to 1 mg) 250-mL standard-taper flat-bottom flask or 150-mL beaker, containing a small boiling aid.

6.6 *Method A*—Filter the dried ether extract, portionwise through a rapid, low-ash paper by decanting from the top opening of the separatory funnel into the appropriate receiver. Cover the filter funnel with a watch glass between each filtration step.

6.7 *Method B*:

6.7.1 Invert the separatory funnel and open the stopcock, allowing any water in the bore of the stopcock to run back into the funnel.

NOTE 3—A small amount of ether applied to and evaporated from the outside of the funnel before the cock is opened will assure that the water will drain back into the funnel.

6.7.2 Close the cock and then shake the contents of the funnel sufficiently for the Na_2SO_4 to absorb all the water. If needed, add more Na_2SO_4 and gently swirl the funnel and contents. Swab out the stem of the funnel with a piece of cotton attached to a stiff wire.

NOTE 4—A small brass screw soldered to the end of a stiff brass wire will serve for this purpose.

6.7.3 Insert a clean plug of cotton moistened with ether into the funnel stem and filter the ether solution through the cotton plug into the appropriate receiver.

NOTE 5—Experience will show how tightly packed the cotton must be to hold back the Na_2SO_4 and still allow a sufficient flow of ether extract.

6.8 Evaporate the ether portionwise from the beaker or distill it from the flask on the steam bath, in a hood. Apply a blanket of nitrogen over the beaker during evaporation. Remove the last portions of fatty acids from the Na_2SO_4 in the funnel by washing with successive small portions of ether until a colorless extract is obtained. Remove the final traces of fatty acids from the filter paper or cotton plug by using several successive small portions of ether.

6.9 If a gas chromatographic analysis of the separated fatty acids is to be performed, observe the precautions given in 7.2 of Test Method D 2245.

6.10 Complete the evaporation of the fatty acid-ether solution on the steam bath, while maintaining a nitrogen atmosphere over the acids. Remove the final traces of ether by heating for successive 20-min periods in a vacuum oven at 60°C until minimum weight is obtained. After each heating period, allow the beaker and contents to cool in a desiccator and then weigh to 1 mg.

7. Calculation and Report

7.1 Calculate the percent total fatty acids content, *T*, as follows, and report the results to 0.1 %:

$$T = \frac{A - B}{S} \times 100 \quad (1)$$

where:

A = weight of beaker plus residue,
B = weight of beaker, and
S = specimen weight taken for analysis.

8. Precision and Bias

8.1 On the basis of an interlaboratory study of this test method, the within-laboratory standard deviation was found to be 0.31 and the between-laboratories standard deviation was found to be 0.50. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.1.1 *Repeatability*—Two results obtained by same operator should be considered suspect if they differ by more than 0.9 % absolute.

8.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 1.5 % absolute.

8.2 *Bias*— No bias can be determined for this test method since no standard alkyd resin exists.

9. Keywords

9.1 alkyd resin; fatty acid; fatty acid content