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Standard Test Method for Approximate Acidity in Electrical Insulating Liquids by Color-Indicator Titration[1]

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1. Scope

1.1 This test method describes the determination of the approximate total acid value of used electrical insulating liquids having viscosities less than 24 cSt at 40°C. It is a simple procedure that can be applied in the field. Where a quantitative neutralization value is required, use Test Method D 664 or D 974. These test methods should be applied in the laboratory.

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 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration[2]

D 974 Test Method for Acid and Base Number by Color-Indicator Titration[2]

D 1193 Specification for Reagent Water[3]

3. Summary of Test Method

3.1 To determine whether the acidity is greater or less than a specified arbitrary value, a fixed volume of liquid to be tested is added to a graduated cylinder, together with a small amount of indicator (phenolphthalein) and the appropriate quantity of standard potassium hydroxide solution. The mixture is shaken and allowed to separate. The color of the aqueous layer at the bottom of the container when testing mineral oils, or at the top when testing askarels, determines whether the acidity is less than or greater than the arbitrary value chosen.

3.2 To determine the approximate total acidity, the procedure is the same as described in 3.1 except that the potassium hydroxide solution is added in small increments until the color of the aqueous layer, after shaking and settling, is a faint pink. The volume of standard potassium hydroxide solution used determines the approximate total acid value.

4. Significance and Use

4.1 The approximate acidity of used electrical insulating oils is an estimate of the total acid value of the oil. As acid values increase, oil quality decreases. This is usually due to oxidation of the oil while in service. In general, acidic by-products produce increased dielectric loss, increased corrosivity, and may cause thermal difficulties attributable to insoluble components called "sludge." This test method is adapted to a specific volume of oil; total acid values of 0.05 to 0.5 mg of potassium hydroxide per gram of oil is a range which is functionally significant.

5. Apparatus

5.1 *Graduated Cylinder*, 50-mL, stoppered.

5.2 *Medicine Dropper*, calibrated at 1-mL intervals.

5.3 *Calibrated Pipet*, 1-mL capacity, calibrated to deliver 0.05-mL increments.

6. Reagents and Materials

6.1 *Purity of Reagents*—Use reagent grade chemicals 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.[4] 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 (see Note 1) conforming to Specification D 1193.

NOTE 1—Any grades of water listed in Specification D 1193 are deemed satisfactory for the purpose of this test method.

6.3 *Ethyl or Isopropyl Alcohol*, reagent grade.

6.4 *Phenolphthalein Indicator Solution* (100 g/L)—Dissolve 10 g of phenolphthalein in 100 mL of denatured alcohol by slight warming.

6.5 *Potassium Hydroxide, Standard Solution* (0.031 ± 0.003 *N*)—Prepare a 0.031 ± 0.003 *N* solution of potassium

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[1] This test method is under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.06 on Chemical Test.

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

[3] *Annual Book of ASTM Standards*, Vols 10.01 and 11.01.

[4] *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

hydroxide (KOH) by dissolving solid KOH in a solution of equal volumes of alcohol and water in accordance with the method of preparation and standardization described in Test Method D 974 or Test Method D 664, except for the solvent and the normality.

NOTE 2—As applied to mineral oil, this normality is chosen to simplify the calculation. The 20-mL sample weighs 17.6 g, assuming an average specific gravity of 0.88. With a 17.6-g sample, 10 mL of 0.031 *N* KOH solution corresponds to an acid value of 1. The normality should be checked from time to time in a laboratory. If it is outside the ±0.003 tolerance, due either to evaporation of solvent or precipitation of potassium carbonate (K_2CO_3) formed by reaction with carbon dioxide (CO_2) from the atmosphere, it should be discarded.

The specific gravity of commonly used askarels is 1.56, or approximately 1.8 times that of mineral oil, and this difference must be recognized in the calculation or equipment calibration, or both, as applied to askarels.

7. Procedure

7.1 Rinse the graduated cylinder first with denatured alcohol and then with a small amount of the liquid to be tested. Pour in the test specimen to the 20-mL mark and add two drops of indicator solution with a medicine dropper. Proceed in accordance with 7.2 or 7.3.

7.2 To determine whether the acidity is greater or less than a value specified by the user, add the appropriate volume of KOH solution by dispensing from a calibrated pipet (for example, 0.5 mL if the fixed figure is 0.05 mg KOH per gram of oil, as applied to oil). Shake vigorously for several seconds, allow to settle, and observe the aqueous layer. If it is free of pink, the acidity is greater than the specified value.

7.3 To determine the approximate total acidity, add KOH solution in increments of 0.5 mL until the color of the aqueous layer, after shaking and settling, is a pale pink.

8. Calculation

8.1 For mineral oil, calculate the approximate total acid value, *T*, as follows:

$$T = (B - A)/10 \quad (1)$$

where:

A = test specimen used, (20) mL,
B = liquid in the cylinder at the conclusion of the test, mL, and
$B - A$ = KOH solution added, mL.

8.2 For askarel, calculate the approximate total acid value, *T*, as follows:

$$T = (B - A)/18 \quad (2)$$

NOTE 3—Other concentrations of base may be used in the titration. A less concentrated solution of KOH provides a greater volume of the aqueous layer. The calculations must then be modified to account for the difference in the concentration of base.

NOTE 4—As an alternative, prepackaged field kits have been used.[5]

9. Report

9.1 Report the following information:

9.1.1 The approximate total acidity value, *T*, in mgKOH/g test specimen.

9.1.2 When possible, report the acidity as “greater than” or “less than” the value specified by the user.

10. Precision and Bias

10.1 This test method provides for an approximation of total acidity, with a minimum detection level of 0.005 mgKOH/g test specimen. The total acidity is based on whether the acidity is greater or less than a user-specified value corresponding to the addition of specific volumes of KOH solution. Because this test method is only an approximation of total acidity, precision and bias statements will not be developed.

10.2 To determine quantitative acidity values, use Test Method D 664 or D 974.

11. Keywords

11.1 acidity; color titration; insulating liquid; neutralization number

[5] Field kits, available from Gerin Co., 1109 Seventh Ave., Neptune, NJ 07753 have been found satisfactory for this purpose.

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