Designation: D3444 – 00 (Reapproved 2015)

Standard Test Method for Total Acid Number of Trichlorotrifluoroethane[1]

This standard is issued under the fixed designation D3444; 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 determination of total acidity in trichlorotrifluoroethane and other halocarbons that are liquid at room temperature.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.* Specific precautionary statements are given in Section 6.

2. Summary of Test Method

2.1 The total acidity of the solvent is determined by titration with standard alcoholic potassium hydroxide solution using phenolphthalein solution as the indicator.

3. Significance and Use

3.1 This test method determines the total acidity of trichlorotrifluoroethane by titration with alcoholic potassium hydroxide using phenolphthalein for an indicator.

3.2 This test method can be used to develop production and purchasing specifications.

4. Apparatus

4.1 *Buret,* 2-mL in 0.01-mL subdivisions, self-leveling, with 1000-mL reservoir.

4.2 *Vinyl Tubing.*[2]

4.3 *Two Indicating Carbon Dioxide Absorbent Tubes, 30–50 mesh.*

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 halide-free distilled water.

5.3 *Isopropyl Alcohol.*

5.4 *Phenolphthalein Indicator Solution* (0.05 g in 100 mL of isopropyl alcohol).

5.5 *Potassium Acid Phthalate,* U.S. National Bureau of Standards primary standard. Dry this chemical in a 120°C oven for several hours prior to use. Store the dry chemical in a desiccator.

5.6 *Potassium Hydroxide Pellets.*

5.7 *Potassium Hydroxide Solution in Isopropyl Alcohol* (0.01 *N*)—Add approximately 1 g of solid KOH to approximately 1 L of anhydrous isopropyl alcohol in a 2-L Erlenmeyer flask. Boil the mixture gently for 5 to 10 min, stirring to prevent the solids from forming a cake on the bottom. Add at least 2 g of barium hydroxide [$Ba(OH)_2$] and boil again gently for 5 to 10 min. Cool to room temperature. Allow to stand for several hours and filter the supernatant liquid through a fine sintered glass or porcelain filtering funnel. Avoid unnecessary exposure to CO_2 during filtration. Store the solution out of contact with cork or rubber. Protect the solution from CO_2 by a guard tube containing carbon dioxide adsorbent. Standardize against pure potassium acid phthalate in about 25 mL of CO_2-free water using phenolphthalein to detect the end point.

6. Hazards

6.1 Trichlorotrifluoroethane removes natural oils from skin. Avoid skin contact. The solvent is not flammable and has a threshold limit value of 1000 ppm by volume in air. See the manufacturer's MSDS.

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[1] This test method is under the jurisdiction of ASTM Committee D26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.04 on Test Methods.

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[2] Tygon has been found satisfactory. An equivalent may be used.

[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 Isopropyl alcohol is flammable, and has a threshold limit value of 400 ppm by volume in air. See the manufacturer's MSDS.

6.3 Potassium hydroxide is corrosive to body tissue. Avoid contact with the solid and its solution. Should this chemical contact the person, immediately flush the affected area with copious amounts of water. See the manufacturer's MSDS.

7. Procedure

7.1 For sample analysis, add 50 mL of isopropyl alcohol to a 250-ml Erlenmeyer flask, add 5 drops of phenolphthalein indicator solution and titrate to a pink end point with alcoholic KOH solution.

7.1.1 Swirl flask contents gently during titration. The end point should last for 10 to 15 s. Stopper the flask until the sample is added to minimize CO_2 contamination.

7.2 Pour a weighed sample (about 50.00 ± 0.02 g) into the flask containing the pretitrated alcohol.

7.3 Titrate the sample with the standard alcoholic KOH solution to a 15-s pink end point. Record the amount of titrant required.

7.4 For standardization of 0.01 *N* KOH in isopropyl alcohol, proceed as follows:

7.4.1 Weigh (±0.0001 g) 0.142 ± 0.005 g of dry (National Bureau of Standards) primary standard potassium acid phthalate into a 1-L volumetric flask containing about 500 mL of deionized CO_2-free water, then stopper the flask.

7.4.2 Swirl the flask to dissolve the potassium acid phthalate.

7.4.3 Fill the flask to volume with deionized, CO_2-free water, stopper the flask then mix the solution thoroughly.

7.4.4 Place 25 mL (transfer pipet) of deionized, CO_2-free water and 5 drops of phenolphthalein indicator solution into a 250-mL Erlenmeyer flask.

7.4.5 Pipet 25 mL (transfer pipet) of the potassium acid phthalate standard solution into the flask, then seal the flask with a stopper.

NOTE 1—Neoprene has been found satisfactory. An equivalent may be used.

7.4.6 Treat the blank and standard alike from this point on.

7.4.7 Titrate the standard with the approximate 0.01 *N* potassium hydroxide solution to the phenolphthalein indicator end point. Record the titer.

8. Calculation

8.1 Calculate the acid number of the solvent as follows:

$$\text{Total acid number in 1 mg of KOH/g sample} = (A \times N \times 56.1)/W \quad (1)$$

where:

A = millilitres of KOH solution required for titration of sample, mL,
N = normality of KOH solution, and
W = weight of sample, g.

8.2 Calculate the normality of the potassium hydroxide standard as follows:

$$N, KOH = (G \times V)/[204.224 \times (A - B)] \quad (2)$$

where:

A = standard titer, mL,
B = blank titer, mL,
G = potassium acid phthalate, g,
V = potassium acid phthalate aliquot, mL, and
204.224 = potassium acid phthalate/mole, g.

8.3 Average the normality values obtained from triplicate analyses to the nearest 0.0001 unit. Use the average normality for sample calculation.

9. Precision and Bias[4]

9.1 *Repeatability (Single Analyst):* At a mean value of 0.0018 mg-KOH/g the standard deviation of results (each the average of triplicates obtained by the same analyst on two different days) has been estimated to be 0.0010 mg-KOH/g at three degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 0.0045 mg-KOH/g.

9.2 *Reproducibility (Multilaboratory):* At a mean value of 0.0018 mg-KOH/g the standard deviation of results (each the average of triplicates in three different laboratories), obtained by analysts in different laboratories, has been estimated to be 0.0036 mg-KOH/g at two degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 0.0219 mg-KOH/g.

10. Keywords

10.1 acidity; CFC-113; total acid number; trichlorotrifluoroethane

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D26-1011.