



Designation: D 3444 – 00

## Standard Test Method for Total Acid Number of Trichlorotrifluoroethane<sup>1</sup>

This standard is issued under the fixed designation D 3444; 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 ( $\epsilon$ ) 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 *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*.<sup>2</sup>

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.<sup>3</sup> 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)<sub>2</sub>] 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<sub>2</sub> during filtration. Store the solution out of contact with cork or rubber. Protect the solution from CO<sub>2</sub> by a guard tube containing Ascarite II.<sup>4</sup> Standardize against pure potassium acid phthalate in about 25 mL of CO<sub>2</sub>-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.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.04 on Test Methods.

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<sup>2</sup> Tygon has been found satisfactory. An equivalent may be used.

<sup>3</sup> *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.

<sup>4</sup> Ascarite II has been found satisfactory and is available from chemical supply houses.