Designation: D3443 – 00 (Reapproved 2015)

Standard Test Method for Chloride in Trichlorotrifluoroethane[1]

This standard is issued under the fixed designation D3443; 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 U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of chloride 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.*

2. Summary of Test Method

2.1 This test method is based on the determination of ionizable chloride by titration with mercuric acetate solution using *s*-diphenylcarbazone as the indicator.

3. Significance and Use

3.1 This test method determines ionizable chloride that can be aqueously extracted from trichlorotrifluoroethane.

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

4. Apparatus

4.1 *Separatory Funnels,* two 250-mL, with polytetrafluoroethylene stopcocks.

4.2 *Erlenmeyer Flask,* 125-mL.

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.[2] 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 *s-Diphenylcarbazone Solution*—Dissolve 0.5 g of *s*-diphenylcarbazone, in 100 mL of methanol.

5.4 *Silver Nitrate Solution*—Dissolve 8.5 g of silver nitrate ($AgNO_3$) in 500 mL of water.

5.5 *Standard Mercuric Acetate Solution*—Dilute 40 mL of stock mercuric acetate solution, reagent 2, to 1000 mL and adjust the pH to 1.6 with nitric acid. Standardize as follows:

5.5.1 Pipet 10.00 mL of standard sodium chloride solution into a 250-mL Erlenmeyer flask. Add 20 mL of chloride-free water and 5 drops of *s*-diphenylcarbazone solution. Titrate with standard mercuric acetate solution to the faint purple end point. Then:

$$\frac{\text{mL NaCl} \times 1.000}{\text{mL mercuric acetate}} = F = \mu\text{g Cl/mL} \tag{1}$$

where

F = factor for the mercuric acetate solution (see 7.1).

5.6 *Standard Sodium Chloride Solution*—Dissolve 0.0660 g of sodium chloride in water and dilute to 1000 mL. Pipet 25 mL of this solution into a 1000-mL volumetric flask, dilute to volume, and mix. One millilitre of this solution contains 1.00 µg of chloride ion.

5.7 *Stock Mercuric Acetate Solution*—Dissolve 1.6 g of mercuric acetate in 500 mL of water containing 3.5 mL of nitric acid. Dilute to 1000 mL and mix.

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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] *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. Procedure

6.1 Wash all glassware with chloride-free water until 10 mL of the washings show no trace of turbidity when 1 mL of silver nitrate solution is added.

6.2 Transfer 100 mL of the sample into a 250-mL separatory funnel, add 25 mL of chloride-free water to the sample and shake for 1 min. Transfer the sample to a second 250-mL separatory funnel and drain the water into a 125-mL Erlenmeyer flask. Repeat the extraction once with another 25 mL of water. Combine the water extracts in the Erlenmeyer flask.

6.3 Warm the water extract to 60°C to drive off any remaining sample. Cool to room temperature.

6.4 Add 5 drops of *s*-diphenylcarbazone solution and titrate with standard mercuric acetate solution to the pale purple end point.

7. Calculation

7.1 Calculate parts per million chloride as follows:

$$\text{Chloride, ppm} = (A \times F)/(S \times G) \quad (2)$$

where:

A = millilitres of mercuric acetate solution required for the titration,
F = factor for the solution (see 5.5.1),
S = millilitres of sample, and
G = specific gravity of the sample.

8. Precision and Bias[3]

8.1 *Repeatability (Single Analyst):* The standard deviation of results (each the average of triplicates obtained by the same analyst on two different days) has been estimated to be 0.005 wt. ppm at four degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 0.02 wt. ppm.

8.2 *Reproducibility (Multilaboratory):* The standard deviation of results (each the average of triplicates in four different laboratories) has been estimated to be 0.06 wt. ppm at three degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 0.3 wt. ppm.

9. Keywords

9.1 CFC-113; chloride; trichlorotrifluoroethane

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[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D26-1000 and RR:D26-1010.