



Designation: D 3448 – 85 (Reapproved 2000)

Standard Test Method for Specific Aqueous Conductance of Trichlorotrifluoroethane¹

This standard is issued under the fixed designation D 3448; 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 approval. 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 aqueous conductivity due to ionizable contaminants extracted from trichlorotrifluoroethane. The value of 1.9 $\mu\text{S}/\text{cm}$ as maximum specific aqueous conductance corresponds to 0.1 ppm of chloride ion (100 ppb). It was calculated by putting the solvent specification of 0.1 ppm chloride into the equation of the subject method to find $C_2 - C_1$ equals 19.08. The specific conductance due to contaminants from the solvent was then calculated as follows:

$$(C_2 - C_1)C_3 = (19)(0.1) = 1.9 \mu\text{S}/\text{cm} \quad (1)$$

1.2 The above is exclusive of the background specific conductance of the water used in the analysis.

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 consult and 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 Ionizable impurities in the solvent are extracted with water of known low conductivity. The increase in water conductivity measures the amount of these impurities present.

3. Significance and Use

3.1 This test method allows for the determination of ionizable contaminants (expressed in terms of ppb of HCl) in trichlorotrifluoroethane via measurement of specific aqueous conductance.

3.2 This test method can be used for establishing manufacturing and purchasing specifications.

4. Apparatus

4.1 Conductivity Bridge.²

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.

Current edition approved May 31, 1985. Published July 1985. Originally published as D 3448 – 75T. Last previous edition D 3448 – 79.

² Serfass Model RCM-15B1 has been found satisfactory and can be obtained from Industrial Instrument Inc., Cedar Grove, NJ. An equivalent may be used.

4.2 *Conductivity Cell*,³ dip-type. Cell constant of 0.1 reciprocal centimetre. Electrodes must be bright platinum.

4.3 *Polyethylene Beaker*,⁴ 250-mL.

4.4 *Polyethylene Bottles*, wide mouth, 300-mL and 500-mL size.

4.4.1 Calibrate and mark the outside of the 300-mL polyethylene bottles to indicate 20, 120, and 220 mL (graduate) of water volume, using deionized water to determine the liquid levels.

4.5 *Polyethylene Tubing*, long enough to reach the bottom of the 300-mL polyethylene bottle.

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.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 *Alcoholic Hydrochloric Acid (0.01 N)*—Dilute 5.0 mL (pipet) of concentrated hydrochloric acid to 6 L with denatured 2B ethyl alcohol.

5.3 *Alumina Gel*.⁶

5.4 *Deionized Trichloromonofluoromethane*, in 50-lb cylinder. This deionized solvent may be selected pure material or it may be purified by multiple passes through an alumina gel contactor (see Fig. 1). Check the acceptability of this deionized solvent as described in 7.4.

NOTE 1—The use of deionized trichloromonofluoromethane will serve as a check on the cleanliness. The deionized standard should be used as

³ Model Cel-A01 has been found satisfactory and can be obtained from Industrial Instrument Inc., Cedar Grove, NJ. An equivalent may be used.

⁴ Catalog No. H-4040, Harshaw Scientific Co., Cleveland, OH, has been found satisfactory. An equivalent may be used.

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.

⁶ Grade F-3 has been found satisfactory and can be obtained from the Aluminum Company of America.

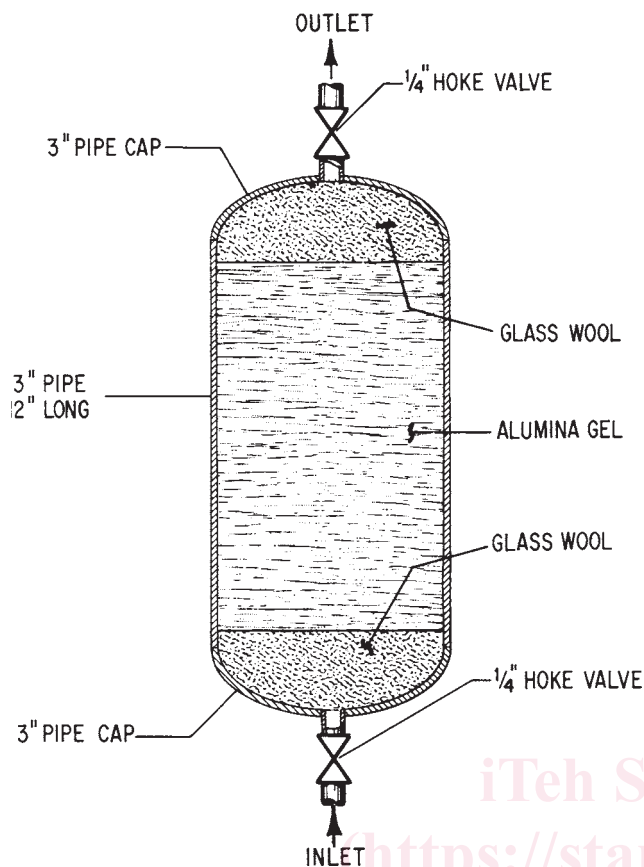


FIG. 1 Alumina Gel Contractor

required on a periodic basis when samples are being evaluated. Standards should be checked whenever high values are obtained or there is doubt of the results.

5.5 *Deionized Water*—Distilled water passed through two mixed resin ion exchangers connected in series and having a conductance of no greater than 0.5 μS . Check the acceptability of the deionized water in accordance with 7.3.

5.6 *Ethyl Alcohol*.

NOTE 2—Specially denatured ethyl alcohol conforming to Formula SD-2B of the U.S. Bureau of Internal Revenue may be substituted for ethyl alcohol (95 %).

5.7 *Hydrochloric Acid*, concentrated.

5.8 *Ion-Exchange Cylinders*.⁷

6. Safety Precautions

6.1 Both trichlorotrifluoroethane and trichloromonofluoromethane are not flammable and have threshold limit value's of 1000 ppm by volume in air. The respective boiling points of these solvents are about 118 and 75°F (47.8 and 23.9°C). Avoid repeated skin contact with the solvents. They remove surface oils from skin.

6.2 Denatured ethyl alcohol is flammable and has a threshold limit value of 1000 ppm by volume in air.

⁷ Ilco-Way ion exchange cylinders, research model, has been found satisfactory and may be obtained from Illinois Water Treatment Co., Rockford, IL.

6.3 *Hydrochloric Acid*—Take all precautions necessary to avoid contact with the body internally and externally. The acid is corrosive.

7. Procedure

7.1 The basis of this test method is the determination by conductance of ions present in the test solution. Therefore, clean all equipment carefully to remove all adsorbed ions in the measuring apparatus, otherwise, erroneously high values will be obtained. Do the initial cleaning and the cleaning of equipment in accordance with 7.2. All equipment must be rinsed with deionized trichloromonofluoromethane before using in a test.

7.2 *Deionizing of Equipment:*

7.2.1 *Polyethylene Bottles and Stainless Steel Cylinders:*

7.2.1.1 Add about 30 g of ion exchange resin from an exchange cylinder to the item to be cleaned. Use an exchange cylinder as a source of resin for cleaning.

7.2.1.2 Add deionized water to the container until it is about two-thirds full.

7.2.1.3 Close the container and roll for at least 24 h on a ball mill roller or its equivalent.

7.2.1.4 Discard the ion-exchange resin and water, rinse with deionized water and then with deionized trichloromonofluoromethane.

7.2.2 *Polyethylene Delivery Tubes:*

NOTE 3—Use of highly plasticized polyethylene tubing and bottles is not recommended.

7.2.2.1 Place the polyethylene delivery tubes in a 500-mL polyethylene bottle.

7.2.2.2 Clean by following 7.2.1.1-7.2.1.4.

7.2.3 *Polyethylene Beakers, Dip-Cell, and Other Equipment:*

7.2.3.1 Add about 30 g of ion-exchange resin to the beaker.

7.2.3.2 Fill the beaker with deionized water.

7.2.3.3 Stir, shake, or transfer the mixture from one container to another for at least 2 min.

7.2.3.4 Discard the ion-exchange resin and deionized water mixture.

7.2.3.5 Rinse the equipment with deionized water and then with deionized trichloromonofluoromethane before using in a test.

7.3 *Preparation of Deionized Water:*

7.3.1 Slowly pass distilled water through two mixed resin ion exchangers, in series, immediately before use, into a clean 250-mL polyethylene beaker. (Note 4). Do not deionize water and store, since it will adsorb carbon dioxide.

NOTE 4—If the conductance of the water is greater than 0.5 μS , repeat the treatment and testing of the water until 0.5 μS or less is obtained, replace the resin exchanger and clean the equipment as described in accordance with 7.2.

7.3.2 Measure the conductivity of the water by the conductivity bridge. Conductance of the water should not be greater than 0.5 μS when measured by a dip-type conductance cell having bright platinum electrodes and a cell constant of 0.1 S.

NOTE 5—The beaker of water used to store the conductance dip-cell should contain deionized water. This water will gradually adsorb carbon