Designation: D3448 – 10 (Reapproved 2015)$^{\varepsilon1}$

Standard Test Method for
Specific Aqueous Conductance of Trichlorotrifluoroethane[1]

This standard is issued under the fixed designation D3448; 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.

ε^1 NOTE—Editorial changes were made in 1.3 and X1.1 in June 2015.

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 µS/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:

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

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

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

1.3.1 *Exception*—Values are stated in inch-pound units in Fig. 1.

1.4 *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]

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

4.3 *Polyethylene Beaker,* 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.[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.

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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] The sole source of supply of the apparatus (Serfass Model RCM-15B1) known to the committee at this time is Industrial Instrument Inc., Cedar Grove, NJ. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[3] The sole source of supply of the apparatus (Model Cel-A01) known to the committee at this time is Industrial Instrument Inc., Cedar Grove, NJ. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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.*[5]

5.4 *Deionized Trichloromonofluoromethane,* in 25-kg 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 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 %).

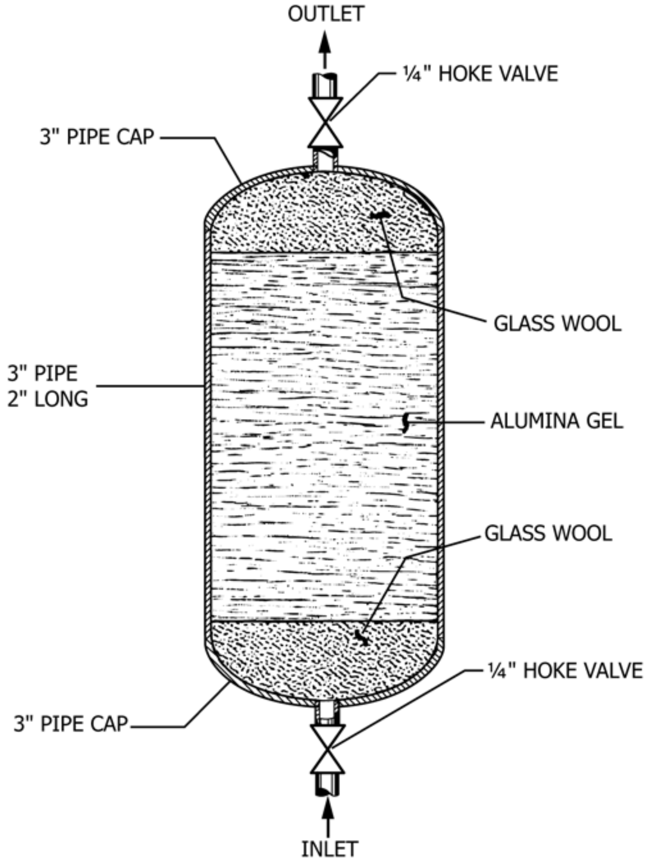

FIG. 1 Alumina Gel Contractor

5.7 *Hydrochloric Acid,* concentrated.

5.8 *Ion-Exchange Cylinders.*[6]

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 47.6 and 23.7°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.

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.

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[5] The sole source of supply of the apparatus (Grade F-3) known to the committee at this time is Aluminum Company of America. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[6] The sole source of supply of the apparatus (Illco-Way ion exchange cylinders, research model) known to the committee at this time is Illinois Water Treatment Co., Rockford, IL. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

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 dioxide, etc. from the air and the conductivity will increase. In order to prevent errors from cell contamination and as long as the apparatus is in constant use, this water should be removed whenever its conductivity exceeds 1.0 µS. During startup, the cell should be dipped in freshly deionized water often enough to remove accumulated ions.

7.4 *Testing of Deionized Trichloromonofluoromethane*—The deionized trichloromonofluoromethane is used to provide a vapor blank to exclude carbon dioxide and other contamination during the analysis.

7.4.1 Add the selected trichloromonofluoromethane to the polyethylene bottle by filling to the 20-mL calibration mark on the bottle. Add the solvent directly from the inverted cylinder without use of any fittings except a cylinder adapter.

7.4.2 Cap the bottle and shake well.

7.4.3 Discard the solvent.

7.4.4 Add another 20 mL of the selected solvent to the bottle.

7.4.5 Immediately add 100 mL of deionized water by filling to the 120-mL calibration mark on the bottle.

7.4.6 Cap the bottle tightly and shake well for 2 min.

7.4.7 Open the bottle and insert the dip-cell of the conductivity bridge into the water layer.

7.4.8 Measure and record conductivity value as the blank conductance.

7.4.9 Remove the dip-cell and place it in a polyethylene beaker of deionized water (see Note 3).

7.4.10 Recap the polyethylene bottle loosely, leaving the solvent and water in the bottle.

7.4.11 Connect a polyethylene delivery tube to the cylinder of selected solvent.

7.4.12 Slowly open the cylinder valve to flush the delivery tube.

7.4.13 Remove the cap from the bottle (7.4.10) and insert the delivery tube so that the tip is below the water level, near the bottom of the bottle.

7.4.14 Add 100 mL of selected solvent (trichloromonofluoromethane) in this manner, filling to the 220-mL calibration mark on the bottle. One hundred millilitre of this solvent is equivalent to 150 g.

7.4.15 Remove the delivery tube and cap the bottle.

7.4.16 Shake the bottle for 2 min, remove the cap, and measure the conductance of the water layer as the second conductance.

7.4.17 Subtract the blank conductance from the second conductance. If a value greater than 0.5 µS/100 mL is obtained, repeat until the value is constant. If a constant value greater than 0.5 µS/100 mL is obtained, the selected trichloromonofluoromethane is not acceptable (see Note 5). The solvent found acceptable will be referred to as deionized trichloromonofluoromethane.

NOTE 6—If a test cylinder has a high conductance value, it will contaminate the equipment. A series of washes with trichloromonofluoromethane of low conductance value will be needed to remove the contamination. The operation is a self-cleaning one, repeated testing of low conductance value solvent will assist in cleaning up of the equipment.

7.5 *Analysis of Trichlorotrifluoroethane:*

7.5.1 Determine the blank conductance of the deionized water and deionized trichloromonofluoromethane as described in 7.4.1 – 7.4.10.

7.5.2 Tare weigh the polyethylene bottle containing the deionized water and deionized trichloromonofluoromethane to the nearest 0.1 g.

7.5.3 Weigh 150 ± 1 g of trichlorotrifluoroethane to the nearest 0.1 g, into the bottle. A volume sample equivalent to 150 ± 1 g may be used instead of a weighed sample.

7.5.4 Recap the bottle tightly and shake it for 2 min.

7.5.5 Open the bottle and measure and record the conductivity value of the water layer as the second conductance.

7.5.6 Loosely cap the bottle. The trichloromonofluoromethane left in the bottle serves to protect the interior from atmospheric contamination. It should be left in the bottle un til a new sample is to be tested at which time it is discarded.

8. Calculation

8.1 Calculate the amount of ionizable contaminants in trichlorotrifluoroethane in terms of ppb of HCl as follows:

$$(C_2 - C_1) \times C_3 \times 52.4 = \text{ppb of soluble ionizable material} \quad (2)$$

calculated as HCl. This value can

be expressed in ppm of HCl or

ppm of chloride.

where:

C_2 = second conductance value (dial reading × multiplier switch setting, measurement of deionized water, deionized trichloromonofluoromethane and sample),

C_1 = blank conductance value (dial reading × multiplier switch setting; measurement of deionized water and deionized trichloromonofluoromethane),

C_3 = cell constant in reciprocal centimetres, and

52.4 = ppb HCl/µS[7,8]

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[7] Lingane, J.J., *Electroanalytical Chemistry*, Interscience Pub., Inc., New York, NY, 1953

[8] Berl, W.G., *Physical Methods in Chemical Analysis*, Vol 11, Academic Press, 125 E. 23rd St., New York, NY, 1951.

9. Precision and Bias

9.1 The precision and bias are estimated to be about ±0.02 µS/cm.

9.2 Repeatability and reproducibility studies are planned.

10. Keywords

10.1 aqueous conductance; conductance

APPENDIX

(Nonmandatory Information)

X1. CALIBRATION OF 52.4 PPB HCl/µS

X1.1 The factor is a theoretical value and can be calculated from data given in Footnotes 7 and 8. The value can be obtained experimentally as follows:

X1.1.1 Determine the blank conductance of the deionized water and deionized trichloromonofluoromethane in accordance with 7.4.1 – 7.4.15.

X1.1.2 Add 0.010 mL of 0.01 *N*alcoholic HCl by syringe into the bottle.

X1.1.3 Cap the bottle and shake it for 2 min.

X1.1.4 Measure and record the conductance value of the water layer as the second conductance.

X1.2 *Calculations*:

$$A/W = 24 \text{ ppb } HCl \text{ in } 100 \text{ m}L \text{ of trichloromonofluoromethane} \quad (X1.1)$$

where:

A = 3.65×10^{-6} g HCl/0.01 mL of 0.01 *N* hydrochloric acid, and

W = 150 g of trichloromonofluoromethane per 100 mL.

$$\frac{24 \text{ ppb HCl}}{(C_2 - C_1)(C_3)} = 52.4 \text{ ppb HCl/}\mu\text{S (using the equipment specified in this test method)} \quad (X1.2)$$

where:

C_2 = second conductance value (dial reading × multiplier switch setting, measurement of deionized water, deionized trichloromonofluoromethane and sample),

C_1 = blank conductance value (dial reading × multiplier switch setting, measurement of deionized water and deionized trichloromonofluoromethane), and

C_3 = cell constant in reciprocal centimetres.

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