



Standard Test Methods for Water-Soluble Halide Ion in Halogenated Organic Solvents and Their Admixtures¹

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1. Scope

1.1 *Test Methods 1, 2, and 3*—These test methods cover the determination of water-extractable halide ion in halogenated organic solvents and their admixtures. Fluoride ion is not measured due to the solubility of silver fluoride.

1.2 *Test Method 4*—This test method covers the determination of chloride ion in halogenated organic solvents and their admixtures.

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 Methods

2.1 *Summary of Test Methods 1, 2, 3*—Halide ion present in halogenated organic solvents is extracted with water and precipitated as the silver halide salt with silver nitrate. Quantity of the halide present is determined by comparing the turbidity of the sample to known standards. The distilled water, as well as all glassware used, must be halide-free.

2.2 *Summary of Test Method 4*—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 These test methods are used to establish manufacturing and purchasing specifications. These test methods will provide a means of determining the condition of the solvent in use. A high water soluble chloride level may indicate the start of solvent decomposition.

4. Apparatus

- 4.1 *Separatory Funnel*, 500-mL,
- 4.2 *Nessler Tubes*, 50-mL,
- 4.3 *Erlenmeyer Flask*, 125-mL, and

4.4 *Colorimeter or Turbidimeter*, Method 2.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all test. 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 specification 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 *Chloride Standard (1 mL \cong 0.0001 g Cl^-)*, *Test Methods 1, 2, and 3*—Prepare by adding 0.165 g of sodium chloride (NaCl) to 1 L of halide-free distilled water.

5.4 *Chloride Standard (1 mL = 0.000001 g Cl^-)*, *Test Method 4*—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.

5.5 *Nitric Acid (sg gr 1.42)*, concentrated nitric acid (HNO_3).

5.6 *Silver Nitrate Solution 0.1 N*—Prepare a 0.1 N solution of silver nitrate ($AgNO_3$).

5.6.1 Prepare the silver nitrate solution by adding 16.99 g of silver nitrate to a 1-L volumetric flask and fill to the line with halide-free distilled water. After shaking to dissolve the $AgNO_3$, store in an amber bottle.

5.7 *Mercuric Acetate Solution*, Test Method 4.

5.7.1 *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.

5.7.2 *Standard Mercuric Acetate Solution*—Dilute 40 mL of stock mercuric acetate solution to 1000 mL and adjust the pH to 1.6 with nitric acid. Standardize in accordance with 7.5.5.

5.7.3 *s-Diphenylcarbazone Solution (Test Method 4)*—Dissolve 0.5 g of *s*-diphenylcarbazone in 100 mL of methanol.

¹ These test methods are under the jurisdiction of ASTM Committee D-26 on Halogenated Organic Solvents and Fire Extinguishing Agents and are 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.