



Standard Test Methods for Water in Halogenated Organic Solvents and Their Admixtures¹

This standard is issued under the fixed designation D 3401; 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 Department of Defense.

1. Scope

1.1 These test methods describe the use of the Karl Fischer (KF) titration for determination of water in halogenated organic solvents and mixtures thereof. Water concentrations from 2 to 1000 ppm can be determined in these solvents. Two test methods are covered as follows:

1.1.1 *Test Method A, Water Determination Using a Coulometric KF Titrator*—The coulometric test method is known for its high degree of sensitivity (typically < 10 µg H₂O) and should be the test method of choice if water concentrations are typically below 50 ppm or if only small amounts of sample are available for water determinations. This test method requires the use of equipment specifically designed for coulometric titrations.

1.1.2 *Test Method B, Water Determination Using a Volumetric KF Titrator*—The volumetric test method is a more traditional approach to KF water determinations. Although titrators are specifically designed for KF volumetric determinations, many automatic titrators on the market can be adapted to perform KF titrations.

1.2 Either of these test methods can be used to determine typical water concentrations (15 to 500 ppm) found in halogenated solvents.

1.3 These test methods recommend the use of commercially available Karl Fischer titrators and reagents.

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 establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see Sections 11 and 15.

2. Referenced Documents

2.1 ASTM Standards:

E 203 Test Method for Water Using Karl Fischer Titration²

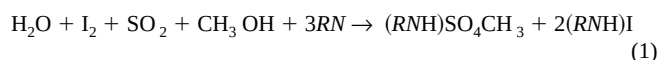
¹ These test methods are under the jurisdiction of ASTM Committee D-26 on Halogenated Organic Solvents and Fire Extinguishing Agents and are the direct responsibility of Subcommittee D26.04 on Test Methods.

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² *Annual Book of ASTM Standards*, Vol 15.05.

3. Summary of Test Methods

3.1 In the Karl Fischer reaction, water will react with iodine in the presence of sulfur dioxide, alcohol, and an organic base according to the following equation:



where RN = organic base.

3.2 When the volumetric titration test method is used for this determination, the halogenated sample is added to a KF solvent that usually consists of sulfur dioxide and an amine dissolved in anhydrous methanol. This solution is titrated with an anhydrous solvent containing iodine. The iodine titrant is first standardized by titrating a known amount of water.

3.3 In the coulometric titration test method, the sample is injected into an electrolytic cell where the iodine required for the reaction with water is produced by anodic oxidation of iodide. With this technique, no standardization of reagents is required.

3.4 In both test methods, the end point is determined amperometrically with a platinum electrode that senses a sharp change in cell resistance when the iodine has reacted with all of the water in the sample.

4. Significance and Use

4.1 High water concentrations can have a detrimental effect on many uses of halogenated solvents.

4.1.1 Water can cause corrosion and spotting when solvents are used for metal cleaning.

4.1.2 Water can reduce the shelf life of aerosol formulations.

4.1.3 Water can inhibit desired reactions when solvents are used in formulations.

5. Interferences

5.1 Certain compounds or classes of compounds interfere with the accurate determination of water by the Karl Fischer test method. They include aldehydes, ketones, free halogens, ferric salts, and strong oxidizing and reducing agents.

5.2 Free halogens can oxidize the iodate in the KF reagents to form iodine; this causes erroneously low water values.