



Designation: E 1064 – 00

Standard Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration¹

This standard is issued under the fixed designation E 1064; 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 This test method covers the determination of water from 0 to 2.0 % in liquid organic chemicals, with Karl Fischer reagent, using an automated coulometric titration procedure.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 Review the current material safety data sheets (MSDS) for detailed information concerning toxicity, first-aid procedures, handling, and safety precautions.

1.4 *This standard does not purport to address all of the safety problems, 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 8.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specifications for Reagent Water²

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals³

E 203 Test Method for Water Using Karl Fischer Reagent³

3. Summary of Test Method

3.1 This test method is based on the Karl Fischer reaction for determining water—the reduction of iodine by sulfur dioxide in the presence of water to form sulfur trioxide and hydriodic acid. The reaction becomes quantitative only when pyridine and methyl alcohol are present. Unlike the volumetric Karl Fischer reagents that include iodine, the coulometric technique electrolytically generates iodine, with 10.71 C of generating current corresponding to 1 mg of water in accordance with Faraday's law.

¹ This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.01 on General Standards.

Current edition approved April 10, 2000. Published July 2000. Originally published as E 1064 – 85. Last previous edition E 1064 – 92.

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 15.05.

4. Significance and Use

4.1 The coulometric technique is especially suited for determining low concentrations of water in organic liquids which would yield small titers by the Karl Fischer volumetric procedure. The precision and accuracy of the coulometric technique decreases for concentrations of water much greater than 2.0 % because of the difficulty in measuring the small size of sample required. The test method assumes 100 % efficiency of coulombs in iodine production. Provision is made for verifying this efficiency.

5. Interferences

5.1 Interfering substances are the same as are encountered in the volumetric Karl Fischer titration and include free halogens, ketones, aldehydes, most acids, and oxidizing or reducing agents. A detailed discussion of interfering substances can be found in the treatise on "Aquametry."⁴

5.2 Test Method E 203 discusses organic compounds in which water may be determined directly and compounds in which water cannot be determined directly, but in which interferences may be eliminated by suitable chemical reactions.

6. Apparatus

6.1 *Automatic Titrator*,^{5,6,7} consisting of a control unit, titration vessel, dual platinum sensing electrode, generator assembly, and magnetic stirrer. The instrument is designed to coulometrically generate iodine that reacts stoichiometrically with the water present in the sample solution. The coulombs of electricity required to generate the reagent are converted to micrograms of water, which is obtained as a direct digital readout.

⁴ J. Mitchell, Jr. and D. M. Smith, "Aquametry"—A Treatise on Methods for the Determination of Water, Part III—The Karl Fischer Reagent, 2nd Ed., J. Wiley and Sons, Inc., New York, NY 1990.

⁵ An apparatus of this type is patented by the Photovolt Corporation under U.S. Patent 3,726,778. A detailed drawing is available from ASTM Headquarters. Request Adjunct No. 12-415330-00.

⁶ The Mitsubishi automatic moisture titrator distributed by Cosa Corporation is patented under U.S. Patent 3,950,237.

⁷ Basic references to the automatic coulometric titrator: M. T. Kelley, R. W. Stelzner, W. R. Laing, and D. J. Fisher, *Analytical Chemistry* 31, No. 2, 220 (1959) and A. W. Meyer, Jr. and C. M. Boyd, *Analytical Chemistry* 31, No. 2, 215 (1959).