



Designation: D1364 – 02 (Reapproved 2007)

## Standard Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)<sup>1</sup>

This standard is issued under the fixed designation D1364; 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 ( $\epsilon$ ) 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 in volatile solvents and chemical intermediates used in paint, varnish, lacquer, and related products.

1.2 This test method is not applicable in the presence of mercaptans, peroxides, or appreciable quantities of aldehydes or amines.

1.3 For purposes of determining conformance of an observed or a calculated value using this test method to relevant specifications, test result(s) shall be rounded off “to the nearest unit” in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E29.

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

1.5 *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.*

1.6 For hazard information and guidance, see the supplier’s Material Safety Data sheet.

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

### 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *instrumental end point, n*—that point in the titration when two small platinum electrodes, upon which a potential of 20 to 50 mV has been impressed, are depolarized by the addition of 0.05 mL of Karl Fischer reagent (6 mg of H<sub>2</sub>O per mL), causing a change of current flow of 10 to 20  $\mu$ A that persists for at least 30 s.

NOTE 1—This end point is sometimes incorrectly called the “dead stop” which is the reverse of the above.

3.1.2 *color end point, n*—During the titration, the solution first turns yellow, then later deepens towards the end of the titration; the end point is indicated by the change from yellow to orange-red which is quite sharp and easily repeated. The orange-red color must persist for at least 30 s in order to indicate an end point.

NOTE 2—View the color by transmitted daylight or by transmitted light from an artificial daylight lamp, such as the one that complies with the specifications given in Test Method D1500.

### 4. Summary of Test Method

4.1 This test method is based essentially upon the reduction of iodine by sulfur dioxide in the presence of water. This reaction can be used quantitatively only when pyridine and an alcohol are present to react with the sulfur trioxide and hydriodic acid produced according to the following reactions:



4.2 To determine water, Karl Fischer reagent (a solution of iodine, pyridine, and sulfur dioxide, in the molar ratio of 1 + 10 + 3) dissolved in anhydrous 2-methoxyethanol is added to a solution of the sample in anhydrous pyridine-ethylene glycol (1 + 4) until all water present has been consumed. This is evidenced by the persistence of the orange-red end-point color; or alternatively by an indication on a galvanometer or similar current-indicating device which records the depolarization of a pair of noble-metal electrodes. The reagent is standardized by titration of water.

\*A Summary of Changes section appears at the end of this standard

