



Standard Test Method for Water in Paints and Paint Materials by Karl Fischer Method¹

This standard is issued under the fixed designation D4017; 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 U.S. Department of Defense.

1. Scope

1.1 This test method is applicable to all paints and paint materials, including resins, monomers, and solvents, with the exception of aldehydes and certain active metals, metal oxides, and metal hydroxides. While the evaluation was limited to pigmented products containing amounts of water in the 30 to 70 % range, there is reason to believe that higher and lower concentrations can be determined by this test method.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 hazard statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

D3960 Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³

E203 Test Method for Water Using Volumetric Karl Fischer Titration

2.2 Other Standard:

EPA Federal Reference Method 24 Determination of Volatile Matter Content, Density, Volume Solids, and Weight

¹ 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 D01.21 on Chemical Analysis of Paints and Paint Materials.

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² 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.

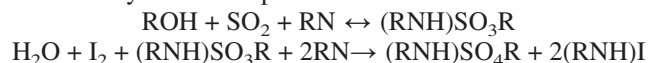
³ The last approved version of this historical standard is referenced on www.astm.org.

Solids of Surface Coatings⁴

3. Summary of Test Method

3.1 The material is dissolved in a suitable solvent, and titrated directly with standardized Karl Fischer reagent, to an electrometric end point. The sluggish reaction with water in pyridine is accelerated with a chemical catalyst, 1-ethylpiperidine.

3.1.1 Karl Fischer reagent is a mixture of iodine, amine, sulfur dioxide, and an alcohol. In the reaction with water, iodine is reduced to hydrogen iodide. Once all the water is consumed, the appearance of free iodine is detected electrochemically and the titration is stopped. The following depicts the chemistry that takes place:



3.2 In classical Karl Fischer titrations the base used is pyridine, and the solvent either methanol or methoxy ethanol. In order to accelerate the reaction when pyridine is used, 1-ethylpiperidine is used as a catalyst/buffer. The additional buffer capacity is usually already built in to most nonpyridine based reagents such as hydranal (see Hydranal Manual).⁵

4. Significance and Use

4.1 Control of water content is often important in controlling the performance of paint and paint ingredients, and it is critical in controlling volatile organic compound (VOC) content.

4.2 Paint materials are often insoluble in common Karl Fischer solvents such as methanol. Pyridine has been found to be a nearly universal solvent for these materials; however, the Karl Fischer reaction is too slow in that solvent at room temperature. To speed it up, 1-ethylpiperidine is added at 5 % as a buffer, or “catalyst.”

4.3 For nonpyridine-based reagents, a number of different solvent systems are available to increase solubility and to minimize interferences from ketones and aldehydes.

⁴ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

⁵ Available from Hoechst Celanese Corporation, Hydranal Technical Center, U.S. Highway 43, Bucks, AL 36512.