



Designation: D1631 – 24

## Standard Test Method for Water in Phenol and Related Materials by the Karl Fischer Reagent Method<sup>1</sup>

This standard is issued under the fixed designation D1631; 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 U.S. Department of Defense.*

### 1. Scope\*

1.1 This test method covers the determination of water in phenol and related materials such as cresols, xylenols, naphthalene, pyridine, and quinoline.

1.2 This test method has been found applicable to a variety of materials varying in water content from 100 mg/kg to solutions containing a relatively high percent of water.

1.3 In determining the conformance of the test results using this method to applicable specifications, results shall be rounded off 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 9.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

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

D1152 Specification for Methanol (Methyl Alcohol)

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.02 on Oxygenated Aromatics.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [www.astm.org/contact](http://www.astm.org/contact). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D1193 Specification for Reagent Water

D3852 Practice for Sampling and Handling Phenol, Cresols, and Cresylic Acid

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

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

E203 Test Method for Water Using Volumetric Karl Fischer Titration

#### 2.2 Other Document:<sup>3</sup>

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200 – Air Contaminates – Table of Exposure Limits and Hazard Communication

### 3. Terminology

3.1 See Terminology D4790 for definition of terms used in this test method.

#### 3.2 Definitions:

3.2.1 *volumetric titration, n*—in reference to Karl Fischer titration methods, a process of measuring the water content of a sample by the physical delivery of a titration reagent containing iodine.

### 4. Summary of Test Method

4.1 This test method is based on the Karl Fischer (KF) reaction for determining water. Iodine is consumed by water in a one to one molar ratio in the presence of sulfur dioxide, organic base, and methanol or other alcohols.

### 5. Significance and Use

5.1 This test method is particularly useful for determining small amounts of water in hygroscopic materials. This test method is suitable for setting specifications on materials

<sup>3</sup> Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, <http://www.access.gpo.gov>.

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

referenced in the scope. It may also be used as an internal quality control tool and in development or research work.

## 6. Interferences

6.1 A number of functional groups are known to interfere with Karl Fischer titrations. In hydrocarbons, the most common interferences are mercaptans and sulfides. Aldehydes and ketones are known to interfere with the Karl Fischer reagent. Some interferences can be mitigated with the use of applicable reagents. For phenol and aromatic compounds, the magnitude of the interference should be negligible under most circumstances. A list of several additional functional groups that can interfere with Karl Fischer titrations is included in Test Method [E203](#).

## 7. Apparatus

### 7.1 Automatic Titrator:

7.1.1 *Volumetric Automatic Titrator*, consisting of a control unit, titration vessel, dual platinum sensing electrode, dispensing buret, and magnetic stirrer. The instrument is designed to accurately dose an iodine containing titrant into the titration vessel that reacts stoichiometrically with the water present in the sample solution. The titrant solution is standardized to determine milligrams of water per milliliter of Karl Fischer reagent it will neutralize in the sample.

7.2 *Gas-tight Syringe*, fitted with a cannula needle of appropriate length and gauge for introducing sample or standard into the titration chamber. The syringe shall be made of glass or other suitably inert material. The volume of the syringe will depend on the sample size. When injecting by volume, the sample should occupy at least 25 % of the syringe volume.

7.2.1 Rinse all glass syringes and needles with dry methanol or ethanol after cleaning, then dry in an oven at 100 °C for at least 1 h and store in a desiccator.

7.3 *Sample Bottle*, suitable for collecting sample and maintaining an air-tight enclosure to prevent intrusion of atmospheric moisture.

7.4 *Oven*, temperature 100 °C  $\pm$  6 °C.

7.5 *Desiccator*, standard laboratory type with desiccant containing color change indicator.

7.6 *Analytical Balance*, capable of weighing to 0.0001 g.

## 8. Reagents

8.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. 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.<sup>4</sup>

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall mean reagent water as defined by Type II and III of Specification [D1193](#).

8.3 *Karl Fischer Reagents*—Pyridine-free formulations are preferred by most KF instrument manufacturers for use with their equipment. These reagents are less toxic, less odorous, and more stable than those containing pyridine. The use of pyridine-free reagents is recommended.

8.3.1 *One-component Karl Fischer Titrant*—Typically consists of a mixture of an organic base, sulfur dioxide and iodine dissolved in a solvent such as diethyleneglycol monoethyl ether. Reagents with titers of H<sub>2</sub>O at 2.0 mg/mL are recommended for this method. Reagents with titers of H<sub>2</sub>O at 5.0 mg/mL may also be used. See [Note 1](#).

8.3.2 *Two-component Karl Fischer Titrant and Solvent*—Titrant typically consists of iodine and methanol or ethanol. Contains sulfur dioxide and organic base in anhydrous methanol or ethanol. See [Note 1](#).

NOTE 1—The “one-component” KF volumetric titrant contains all the necessary reagents for titration of water and the solvent is used basically as a medium to dissolve the sample. The “two-component” KF volumetric titrant contains iodine and methanol or ethanol and the solvent contains sulfur dioxide and a base. The one component system is simple and allows for flexibility in solvent choice. However, because all of the necessary chemicals for titration are present, the titer decreases significantly over time. The two-component system will remain stable for longer periods of time and can provide more accurate measurement of trace water concentrations. Two-component reagents, however, are more susceptible to side reaction from non-complexed sulfur dioxide than single-component systems.

### 8.4 Solvents:

8.4.1 *Methanol*, max 0.15 % water, in accordance with Specification [D1152](#). (See [Note 2](#).)

NOTE 2—Solvents with low water content will consume less Karl Fischer reagents during the pre-titration process.

8.5 *Water Standard*—Solvent mixture with precisely determined water content intended for calibrating volumetric KF reagents. Standards should come complete with a Certificate of Analysis. Standards containing 1.0 % water are recommended for this test method. Type II water may also be used for titrant standardization.

## 9. Safety Precautions

9.1 The reagents contain one or more of the following: iodine, organic base, sulfur dioxide, and methanol or other

<sup>4</sup> *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.