Designation: D1631 – 10 (Reapproved 2018)ε1

Standard Test Method for Water in Phenol and Related Materials by the Iodine Reagent Method[1]

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 (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.

ε[1] NOTE—Section 14 was editorially corrected in February 2018.

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:*[2]

D1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)

D3437 Practice for Sampling and Handling Liquid Cyclic Products

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Document:*[3]

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

3. Terminology

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

4. Summary of Test Method

4.1 When solutions of iodine in methanol and of sulfur dioxide in pyridine are mixed in the presence of water, the following reaction occurs:

$$I_2 + SO_2 + H_2O \rightleftarrows 2HI + SO_3 \quad (1)$$

4.1.1 Sufficient pyridine is present in the reagent to consume the hydriodic acid and sulfur trioxide:

[1] 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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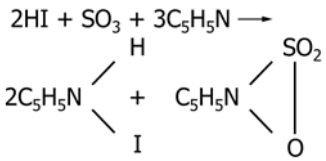
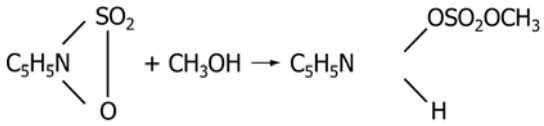
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] 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



4.1.2 The pyridine sulfur trioxide salt reacts with the methanol, this preventing a second mole of water from being consumed:



4.2 When the pyridine solution contains water and the sulfur dioxide is titrated with iodine in methanol solution, the platinum electrodes remain polarized until all the water reacts. A slight excess of iodine depolarizes the electrodes, allowing current to flow through the microammeter which indicates the end point.

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

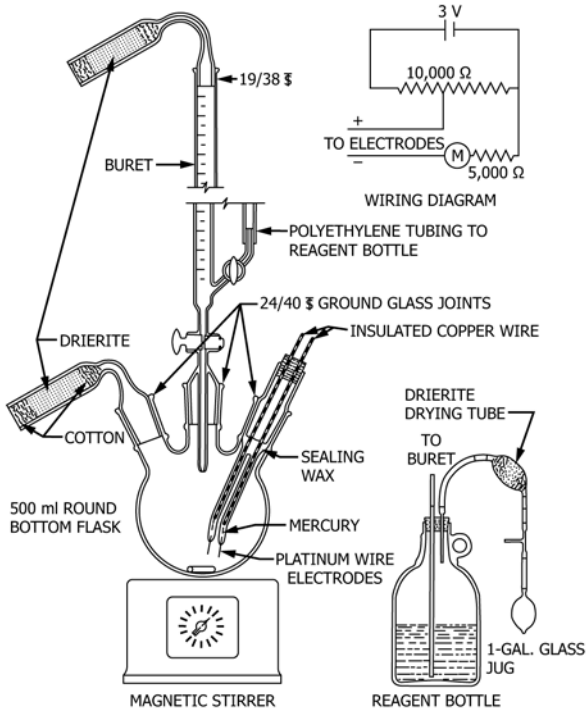


FIG. 1 Titration Assembly

6.2 If ketones are present in the sample, interference from them can be avoided by employing the glycol-pyridine sample solvent specified in Test Method D1364.

7. Apparatus

7.1 The apparatus shall be assembled as shown in Fig. 1. Any suitable modification permitting equal facility and accuracy may be used. Automatic titration equipment is commercially available and may be used.

8. Reagents

8.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] 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.

8.2 *Iodine Solution*—Dissolve 150 g of iodine (I_2) crystals in 3 L of anhydrous methanol. Place the solution in the reagent bottle connected to the buret as shown in Fig. 1.

8.3 *Methanol,* anhydrous, containing less than 0.05 % water.

8.4 *Pyridine Solution*—Place 4000 mL of refined grade pyridine in a 5000-mL distilling flask. Distill over and discard 400 mL of forecut at atmospheric pressure. Distill off 3400 mL of center cut and transfer to a suitable glass bottle fitted with a two-hole stopper. Through one hole of the stopper insert a piece of glass tubing that extends almost to the bottom of the bottle; through the other hole insert a short piece of glass tubing to serve as a vent. Through the long tube add 400 g of refrigerant-grade sulfur dioxide (SO_2) dried through concentrated sulfuric acid (H_2SO_4 sp gr 1.84), and allow the solution to cool. Fit the vent tube with a drying tube and an aspirator bulb; connect the long tube with an adapter suitable for introducing the reagent into the titration flask. For convenience in measuring, a suitable reservoir may be placed in the system.

NOTE 1—In place of the divided reagents described in 8.2, 8.3, and 8.4 it is permissible to employ the single solution reagent specified in Test Method D1364 or commercial Karl Fischer reagents. Pyridine-free reagents are available from various laboratory suppliers and may be used if suitable for the material being tested.

8.5 When handling Karl Fischer reagent refer to Practice D3437.

9. Hazards

9.1 Consult current OSHA regulations supplier's Safety Data Sheets and local regulations for all materials used in this test method.

[4] *Reagent Chemicals, American Chemical Society Specifications*, 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.