



Designation: D 1631 – 99

Standard Test Method for Water in Phenol and Related Materials by the Iodine Reagent Method¹

This standard is issued under the fixed designation D 1631; 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.

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 The following applies to all specified limits in this standard: for purposes of determining conformance with this standard, an observed value or a calculated value 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 E 29.

1.4 *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.* For specific hazard statements, see Section 9.

2. Referenced Documents

2.1 ASTM Standards:²

D 1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)

D 3437 Practice for Sampling and Handling Liquid Cyclic Products

D 3852 Practice for Sampling and Handling Phenol and Cresylic Acid

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals²

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³

2.2 Other Document:⁴

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.02 on Oxygenated Aromatics.

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² Annual Book of ASTM Standards, Vol 06.04.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

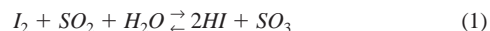
OSHA Regulations, 29 CFR, Paragraphs 1910.1000 and 1910.1200

3. Terminology

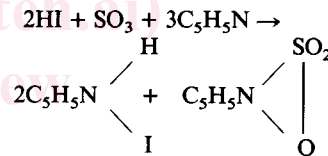
3.1 See Terminology D 4790 for definition of terms used in this test method.

4. Summary of Test Method

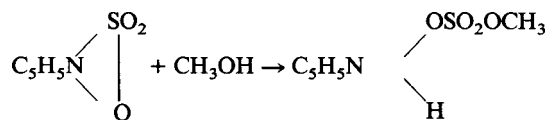
4.1 When solutions of iodine in methanol and of sulfur dioxide in pyridine are mixed in the presence of water, the following reaction occurs:



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



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