



Standard Test Method for Pyridine Bases in Cresylic Acid by Direct Titration¹

This standard is issued under the fixed designation D4471; 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 pyridine and other basic nitrogen impurities in crude and refined cresylic acids streams, including mixtures.

1.2 This test method is applicable for pyridine base levels of 0.001 % to 0.5 %.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 8.

2. Referenced Documents

2.1 ASTM Standards:²

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

2.2 Other Document:

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200³

3. Terminology

3.1 For definitions of terms used in this test method see Terminology D4790.

4. Summary of Test Method

4.1 This test method is a direct, nonaqueous titration technique utilizing perchloric acid in acetic acid as titrant and the cresylic acid itself as titration solvent. Endpoints may be established potentiometrically as well as by indicator so that the method is applicable to highly colored as well as lighter colored materials. This test method will detect basic components other than pyridine bases should they be present. All basic compounds detected by this procedure are calculated and expressed as percent pyridine.

5. Significance and Use

5.1 The pyridine base content of cresylic acids is important in certain applications. This test method may be used as a tool for quality control and specification purposes by producers and users.

6. Apparatus

6.1 *Titrimeter or pH meter*, equipped with half cell or combination glass pH and Ag/AgCl reference electrodes. The pair of electrodes shall be mounted to extend well below the liquid level. Storage in water between titrations is essential because prolonged immersion in nonaqueous medium significantly deadens response.

6.2 *Buret*, 50-mL capacity.

6.3 *Magnetic Stirrer*, with TFE-fluorocarbon or glass covered stirring bar.

6.4 *Autotitration Equipment* may be used if available.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

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 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, Mail Stop: SDE, Washington, DC 20401.

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