



## Standard Test Method for Pyridine Bases in Cresylic Acid by Direct Titration<sup>1</sup>

This standard is issued under the fixed designation D 4471; 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 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 8.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 3852 Practice for Sampling and Handling Phenol and Cresylic Acid<sup>2</sup>

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals<sup>2</sup>

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>3</sup>

#### 2.2 Other Document:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200<sup>4</sup>

### 3. Terminology

3.1 For definitions of terms used in this test method see Terminology D 4790.

### 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 glass and calomel 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 all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>5</sup> 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.

7.2 *Perchloric Acid Titrant* (0.02 N in glacial acetic acid)—

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.04.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

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

<sup>5</sup> *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.