Designation: D1614 – 09 (Reapproved 2023)

Standard Test Method for Alkalinity in Acetone[1]

This standard is issued under the fixed designation D1614; 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.

1. Scope

1.1 This test method covers the determination in acetone of alkalinity calculated as ammonia (NH_3).

1.2 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 E29.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 For specific hazard information and guidance, consult the supplier's Safety Data Sheet.

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.* Specific hazard statements are given in Section 7.

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]

D1193 Specification for Reagent Water

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

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

3. Summary of Test Method

3.1 The specimen is added to water previously neutralized to the methyl red end point. If alkalinity is detected, it is titrated with 0.05 *N* H_2SO_4 and reported as weight percent of NH_3.

4. Significance and Use

4.1 This test method provides a measurement of alkalinity in acetone. The results of this measurement can be used for specification acceptance.

5. Apparatus

5.1 *Buret,* 10 mL, graduated in 0.05 mL subdivisions.

5.2 *Erlenmeyer Flask,* 250 mL capacity.

6. Reagents and Materials

6.1 *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.[3] 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV or higher purity of Specification D1193.

6.3 *Methyl Red Indicator Solution* (1 g/L)—Dissolve 0.1 g of methyl red in 100 mL of methanol, ethanol, or isopropanol. Prepare a fresh solution at least once a month as needed.

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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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] *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.

6.4 *Sodium Hydroxide, Standard Solution* (0.05 *N*)—Prepare and standardize a 0.05 *N* sodium hydroxide (NaOH) solution (Note 1) in accordance with Sections 12 to 17 of Practice E200.

NOTE 1—Alternatively, potassium hydroxide (KOH) solution may be used.

6.5 *Sulfuric Acid, Standard Solution* (0.05 *N*)—Prepare and standardize a 0.05 *N* sulfuric acid (H_2SO_4) solution.

7. Hazards

7.1 Acetone is a highly flammable liquid.

7.2 The reagents sulfuric acid and sodium hydroxide are hazardous as they can cause severe burns of the skin or eyes.

8. Procedure

8.1 To a 250 mL Erlenmeyer flask, add 50 mL of water and 3 drops of methyl red indicator solution. If the water is basic, neutralize to the first faint pink coloration with 0.05 *N* H_2SO_4. If acidic, neutralize to the first yellow coloration with 0.05 *N* NaOH solution and then to the first faint pink coloration with 0.05 *N* H_2SO_4 solution. Now add 50 mL of sample to the neutralized water. If there is no change in the color of the solution, the sample may be considered free of alkalinity. If, however, the solution turns yellow, titrate it with 0.05 *N* H_2SO_4 to the first pink coloration.

9. Calculation

9.1 When it is necessary to titrate the solution with the 0.05 *N* H_2SO_4 calculate the percent of alkalinity as ammonia (NH_3) as follows:

$$NH_3, \% = (VN \times 0.034)/D \quad (1)$$

where:

V = H_2SO_4 required for titration of the specimen, mL,
N = normality of the H_2SO_4, and
D = density of the specimen at the test temperature in g/ml.

10. Report

10.1 If the solution does not turn yellow, report alkalinity as zero.

10.2 If the solution is alkaline, report the percent of ammonia to the nearest 0.0001 %. Duplicate runs that agree within 0.00007 % absolute are acceptable for averaging (95 % confidence level).

11. Precision and Bias[4]

11.1 *Precision:*

11.1.1 On the basis of an interlaboratory study of this test method in which operators in eleven laboratories analyzed one sample of acetone with a mean alkalinity of 0.0009 %, the within-laboratory standard deviation was found to be 0.00002 % absolute with nine degrees of freedom and the between-laboratories standard deviation 0.00007 % absolute with eight degrees of freedom. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1.1 *Repeatability*—Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than 0.0001 % absolute.

11.1.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.0002 % absolute.

11.2 *Bias*—Bias cannot be determined for this test method because there is no available material having an accepted reference value.

12. Keywords

12.1 acetone; alkalinity test

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1020. Contact ASTM Customer Service at service@astm.org.