Designation: D 1614 – 95 (Reapproved 1999)

Designation: D 1614 – 03

Standard Test Method for Alkalinity in Acetone[1]

This standard is issued under the fixed designation D 1614; 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 Department of Defense.

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

Current edition approved ~~Nov.~~ July 10, ~~1995.~~ 2003. Published ~~January 1996.~~ August 2003. Originally ~~published as D 1614 – 58.~~ approved in 1958. Last previous edition approved in 1995 as D 1614 – 95 (1999).

1. Scope*

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

1.2 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

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

1.4 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.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[3]

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[4]

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.[5] 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.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol ~~15.05.~~ 14.02.

[4] ~~*Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing~~Annual Book 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.~~ ASTM Standards, Vol 15.05.

[5] ~~Supporting data are available from ASTM Headquarters. Request RR:D01-1020.~~

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

6.3 *Methyl Red Indicator Solution* (1 g/L)—Dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol. Prepare a fresh solution at least once a month as needed.

6.4 *Sodium Hydroxide, Standard Solution* (0.05 *N*)—Prepare and standardize a 0.05 *N* sodium hydroxide (NaOH) solution (Note) in accordance with Sections 12 to 17 of Practice E 200.

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 = ~~specific gravity~~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 [6]

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; alkalinity test; acetone~~ test

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[5] *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.

[6] Supporting data are available from ASTM International Headquarters. Request RR:D01-1020.

SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D 1614 - 95 (1999)) that may impact the use of this standard.

(1) Added Practice E 29 on significant digits to the scope.
(2) Added Practice E 29 to the Referenced Documents section.
(3) Changed “specific gravity” to “density” and added “g/ml” to the definition of D in Section 9 calculation.