Designation: D2989 – 01 (Reapproved 2016)

Standard Test Method for Acidity-Alkalinity of Halogenated Organic Solvents and Their Admixtures[1]

This standard is issued under the fixed designation D2989; 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 of acidity in halogenated organic solvents and admixtures thereof. The alkalinity may be determined utilizing Test Methods D2106, by substituting the end point measured at pH 7 by bromothymol blue or pH meter.

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

1.3 *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.* Specific precautionary statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2106 Test Methods for Determination of Amine Acid Acceptance (Alkalinity) of Halogenated Organic Solvents

D2110 Test Method for pH of Water Extractions of Halogenated Organic Solvents and Their Admixtures

3. Summary of Test Method

3.1 A sample of halogenated solvent or admixture is measured for pH using Test Method D2110. If the pH of the sample is above 7.0, the alkalinity is determined using Test Methods D2106 (to an end point of pH 7). If the pH is below 7.0, the free acid content of the halogenated organic solvent or admixture is determined after water extraction using Procedure A, or can be determined directly using Procedure B.

3.1.1 *Procedure A,* using glass electrode pH meter, or

3.1.2 *Procedure B,* anhydrous methanolic sodium hydroxide titration.

4. Significance and Use

4.1 This test method can be used to establish manufacturing and purchasing specifications. It can also be used to determine the condition of solvents in use.

5. Apparatus

5.1 *Separatory Funnel,* 250 mL.

5.2 *Graduated Cylinder,* 100 mL.

5.3 *Volumetric Pipets,* 1 mL, 10 mL, 25 mL, 50 mL.

5.4 *Beaker,* 100 mL.

5.5 *Borosilicate or Stainless Steel Beaker,* 2 L.

5.6 *Erlenmeyer Flask,* 100 mL.

5.7 *pH Meter with pH Electrodes.*

5.8 *Buret,* 10 mL.

5.9 *Volumetric Flask,* 100 mL, 1 L.

5.10 *Micro Buret,* 5 mL, Class A or *Syringe*, 100 µL.

6. Reagents

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 *Water*—Prepare neutral, distilled or deionized water as follows: Boil 1 L of distilled or deionized water for 5 min in a

[1] This test method is under the jurisdiction of ASTM Committee D26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.04 on Test Methods.

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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] *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. (USP), Rockville, MD.

borosilicate glass or stainless steel container, then cover and cool to room temperature. Titrate to a pH of 7.0 to 7.3 with either 0.01 *N* sodium hydroxide (NaOH) solution or 0.01 *N* hydrochloric acid (HCl), depending upon whether the initial pH is above or below 7.

6.3 *Bromothymol Blue Indicator Solution*(0.1 %)—Indicator solution may be purchased from a laboratory supply house or prepared as follows: Dissolve 0.1 g of dibromothymol sulfonephthalein in 1.6 mL of 0.1 *N* NaOH solution and dilute to 100 mL with methanol.

6.4 *Sodium Hydroxide Solution* (0.01 *N*), *Procedure A*—Dissolve 4 g of sodium hydroxide (NaOH) in boiled, neutral, distilled or deionized water and dilute to 1000 mL. Prepare 0.01 *N* NaOH solution by diluting 10.0 mL of the 0.1 *N* NaOH to 100 mL with boiled, neutral, distilled or deionized water in a calibrated volumetric flask. Standardize this 0.01 *N* NaOH by any accepted procedure to determine the exact normality. This reagent is also available from most supply houses. Protect the reagent from absorption of atmospheric carbon dioxide and standardize weekly.

6.5 *Sodium Hydroxide Methanolic Solution* (0.01 *N*), *Procedure B*—Dissolve 4 g of ACS reagent grade sodium hydroxide (NaOH) in ACS reagent grade anhydrous methanol and dilute to 1000 mL with ACS reagent grade anhydrous methanol. Prepare 0.01 *N* methanolic NaOH solution by diluting 10.0 mL of the 0.1 *N* NaOH to 100 mL with ACS reagent grade anhydrous methanol in a calibrated volumetric flask. Standardize this 0.01 *N* NaOH by any accepted procedure to determine the exact normality. This reagent is also available from most supply houses. Protect the reagent from absorption of atmospheric carbon dioxide and standardize weekly.

7. Hazards

7.1 *Solvent Hazards*—Contact with skin should be avoided to prevent removal of natural oils. Solvents are not flammable, but each has a threshold limit value for contact with vapors. These threshold limits should be determined before a solvent is listed.

7.2 NaOH as a solid or in solution can be hazardous if there is skin contact.

8. Procedure

8.1 *Procedure A*—Pipet 50 mL of halogenated organic solvent or admixture into a 250-mL separatory funnel containing 50 mL of freshly boiled, neutral (pH 7.0 to 7.3), distilled or deionized water. Shake the mixture for 2 min, then let stand and allow the layers to separate. Drain and discard the lower, organic layer from the separatory funnel and transfer the water layer to a 100-mL beaker. Titrate the sample, with stirring, with 0.01 *N* NaOH using a 5-mL Class A buret until the pH is stable between 7.0 and 7.3 for 30 s. A 100-µL syringe can be substituted for the micro buret. This technique is often used to measure very low levels of acidity, below 1 ppm. Technique is very important. Titrate slowly, making sure the titrant falls cleanly into the water being titrated. Record the titer and calculate the acidity in accordance with 9.1.

8.2 *Procedure B*—Pipet 50 mL of halogenated organic solvent or admixture into a clean, dry, 100-mL Erlenmeyer flask. Add 1 mL of bromothymol blue indicator solution and titrate, with stirring, with 0.01 *N* methanolic NaOH solution to the bromothymol blue end point (see Note 1).

NOTE 1—The bromothymol blue end point may be interpreted as an aqua (blue-green) to light blue color.

9. Calculation

9.1 *Procedure*—Calculate the acidity of halogenated organic solvents or admixtures as equivalent hydrochloric acid in weight percent or weight ppm as follows:

$$\text{equivalent HCl, weight \%} = [(V \times N \times 0.0365) / W] \times 100 \quad (1)$$

$$\text{equivalent HCl, ppm} = [(V \times N \times 0.0365) / W] \times 1\,000\,000$$

where:

V = Volume in mL, NaOH solution required for titration of the sample,
N = Normality of the NaOH solution, and
W = Weight of the sample used, in g as weighed or g = sample volume in mL times its density in g per mL.

10. Precision [4]

10.1 Using Method A, one standard deviation between results of a sample run by different laboratories should be no more than about ±0.5 ppm. Within a laboratory, one standard deviation between results should be no more than about 0.2 ppm. Bias was not determined in this study.

10.2 Using Method B, one standard deviation between results of a sample run by different laboratories should be no more than about ±1.0 ppm. Within a laboratory, one standard deviation between results should be no more than about 0.1 ppm. Bias for this method is approximately +1 ppm.

11. Keywords

11.1 acidity; alkalinity; bromothymol blue; halogenated solvents; hydrochloric acid; pH meter; sodium hydroxide; titration

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D26-1016.