



Standard Test Method for Acidity-Alkalinity of Halogenated Organic Solvents and Their Admixtures¹

This standard is issued under the fixed designation D 2989; 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. 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 Method D 2106, by substituting the end point measured at pH7 by bromothymol blue or pH meter.

1.2 *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:

- D 2106 Test Method for the Determination of Amine Acid Acceptance (Alkalinity) of Halogenated Organic Solvents²
- D 2110 Test Method for pH of Water Extractions of Halogenated Organic Solvents and Their Admixtures²

3. Summary of Test Method

3.1 A sample of halogenated solvent or admixture is measured for pH using Test Method D 2110. If the pH of the sample is above 7.0, the alkalinity is determined using Test Method D 2106 (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 B, or can be determined directly using Procedure C.

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

3.1.2 *Procedure B*, using indicator.

3.2 *Procedure C*, 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.

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 water as follows: Boil 1 L of distilled water for 5 min in a borosilicate glass or stainless steel container, then cover and cool to room temperature. Titrate a 50-mL aliquot 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. From this titer, calculate the volume of 0.01 N NaOH solution or 0.01 N HCl required to adjust the pH of the remaining 950 mL of boiled distilled water to a pH of 7.0 to 7.3. Add the calculated volume of 0.01 N NaOH solution or 0.01 N HCl together with the titrated 50-mL aliquot of neutralized water to the 950-mL stock and mix thoroughly. Determine the pH of a second aliquot. If further adjustment is necessary to obtain a pH of 7.0 to 7.3, repeat this procedure.

6.3 *Bromothymol Blue Indicator Solution (0.1 %)*—Indicator solution may be purchased from a laboratory supply

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² *Annual Book of ASTM Standards*, Vol 15.05.

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. (USPC), Rockville, MD.

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), Procedures A and B*—Dissolve 4 g of sodium hydroxide (NaOH) in boiled, neutral, distilled water and dilute to 1000 mL. Standardize this 0.1 N NaOH by any accepted procedure to determine the exact normality. Prepare 0.01 N NaOH solution by diluting 10.0 mL of the standardized 0.1 N NaOH to 100 mL with boiled, neutral, distilled water in a calibrated volumetric flask. 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 C*—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. Standardize this 0.1 N NaOH by any accepted procedure to determine the exact normality. Prepare 0.01 N methanolic NaOH solution by diluting 10.0 mL of the standardized 0.1 N NaOH to 100 mL with ACS reagent grade anhydrous methanol in a calibrated volumetric flask. 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 water. Shake the mixture for 2 min, then let stand and allow the layers to separate. Pipet a 25-mL aliquot of the water layer into a 100-mL beaker. Titrate the sample, with stirring, until the pH is stable between 7.0 and 7.3 for 30 s. 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 250 mL separatory funnel containing 50 mL of freshly boiled, neutral, distilled water adjusted to a bromothymol blue end point (see Note 1). Shake the mixture

for 2 min, then let stand and allow the layers to separate. Pipet a 25-mL aliquot of the water layer into a 100-mL beaker. Add 1 mL of bromothymol blue indicator solution to the sample. Titrate, with stirring, with 0.01 N NaOH solution to the bromothymol blue end point (see Note 1). Record the titer and calculate the acidity in accordance with 8.1.

8.3 *Procedure C*—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 *Procedures A and B*—Calculate the acidity of halogenated organic solvents or admixtures as equivalent hydrochloric acid in weight percent or weight ppm as follows:

$$\text{equivalent HCl, weight \%} = [V \times N \times 0.0365 \times 2]/W \times 100 \quad (1)$$

$$\text{equivalent HCl, ppm} = [V \times N \times 0.0365 \times 2]/W \times 1\,000\,000$$

where:

V = volume, in mL, NaOH solution required for titration of the sample,

N = normality of the NaOH solution,

W = weight of the sample used, g, (sample volume, mL, times its density),

2 = factor to allow for the fact that only half of the water extract is titrated.

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

$$\text{equivalent HCl, weight \%} = [V \times N \times 0.0365]/W \times 100 \quad (2)$$

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

10. Precision and Bias

10.1 Different laboratories using different instruments should be able to obtain results that differ from the mean by not more than 5 %. The precision and bias of this test method is based on current experience. The results are valid for levels of acidity as HCl greater than 2 ppm for methods A, B, and C.

11. Keywords

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

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