Standard Test Method for Total Acid Acceptance of Halogenated Organic Solvents (Nonreflux Methods)[1]

This standard is issued under the fixed designation D2942; 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 the total acid acceptance including amine and neutral type (alpha epoxide) stabilizers in halogenated organic solvents.

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]

D1193 Specification for Reagent Water
D2106 Test Methods for Determination of Amine Acid Acceptance (Alkalinity) of Halogenated Organic Solvents
E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

3. Summary of Test Method

3.1 *Test Method A*—The total acid acceptance is determined by the reaction of the sample with a known amount of hydrochloric acid that has been dissolved in isopropyl alcohol. The excess acid remaining after the reaction with the acid acceptor is then back-titrated with a standardized solution of sodium hydroxide. The total acid acceptance of the stabilizers can be calculated and reported as weight % NaOH.

3.2 *Test Method B*—The acid acceptor content is determined by gas chromatography or another validated method such as density or refractive index and the acid acceptance is calculated from the weight % acid acceptor. Techniques like density and refractive index are generally suitable only for two-part systems. The method for the determination of the acid acceptor should be validated for the sample being analyzed to ensure there is no interference, particularly from such things as oils and reaction products when measuring acid acceptance of product that is in use such as vapor degreasers.

4. Significance and Use

4.1 Solvents that have been depleted in stabilizer content can become acidic. Acids can cause corrosion to process and storage equipment used for halogenated solvents.

4.2 Halogenated organic solvents may contain amine type (alkaline, aqueous extractable) acid accepting additives, neutral type (typically epoxide) acid accepting additives or both. This test method can determine the combined acid acceptance from both types of stabilizers. In addition, if the amine acid acceptance is determined by a separate procedure (see Test Methods D2106), the amount of neutral acid accepting stabilizers in a solvent can be calculated from the difference between combined and amine acid acceptance values.

4.3 This test method may be used by producers and users to verify that a product is complying with acid acceptance product specifications or by users to monitor the acid accepting ability of a solvent in use.

5. Test Method A

5.1 *Apparatus*

5.1.1 *Iodine or Erlenmeyer Flasks* 250-mL, with ground-glass stoppers.

5.1.2 *Burets,* 25-mL or 50-mL, graduated to 0.1 mL.

5.1.3 *Pipet,* 10-mL and 25-mL.

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,

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[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.

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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 Specification D1193.

6.3 *Bromophenol Blue Indicator Solution* (1.0 g/L)—Prepared by dissolving 1.0 g of bromophenol blue in a mixture of 800 mL of water and 200 mL of denatured ethyl alcohol. The pH range is 3.0 to 4.6. The color change is from yellow on the acid side to blue-green on the alkaline side.

6.4 *Hydrochlorination Reagent* (0.1 *N* HCl)—Prepare by dissolving 4.4 mL of concentrated hydrochloric acid (sp gr 1.19) in isopropyl alcohol and diluting to 500 mL with isopropyl alcohol.

6.5 *Sodium Hydroxide, Standard Solution* (0.1 *N*)—Prepare and standardize in accordance with Practice E200.

7. Safety Precautions

7.1 Solvent Hazard—Contact with the skin should be avoided to prevent the removal of natural oils.

7.2 Observe the published safety procedures when handling the concentrated HCl and NaOH solutions.

8. Procedure

8.1 *Blank*—Pipet 25 mL of hydrochlorination reagent and 25 mL of isopropyl alcohol with 25-mL pipets into a 250-mL Erlenmeyer flask. Add 3 drops of bromophenol blue indicator solution and titrate to a stable blue-green end point with 0.1 *N* NaOH solution. Record the millilitres of NaOH solution required as *A* (see Section 9 for calculation).

8.2 *Sample*—Transfer 25 mL of hydrochlorination reagent by means of a 25 mL pipet to a 250-mL glass-stoppered Erlenmeyer flask. Add 10 mL of dry halogenated solvent and 25 mL of isopropyl alcohol. Shake the mixture thoroughly and allow to stand at room temperature for 10 min. Add 3 drops of bromophenol blue indicator solution to the sample flask and titrate with 0.1 *N* NaOH solution to the blue-green end point. Record the millilitres of NaOH solution required as *B* (see Section 9 for calculation).

NOTE 1—Make sure flask and pipette are *dry* before running analysis. If turbidity or phase separation occurs, add a small amount of isopropanol.

8.3 If the amount of nonamine acid acceptance must be known, first determine the amine acid acceptance in accordance with Test Method D2106.

9. Calculation

9.1 *Total Acid Acceptance*—Calculate the total acid acceptance as equivalent NaOH in weight % as follows:

$$\text{Equivalent NaOH weight, } \% = \left[(A - B)N \times 0.04 \times 100 \right] / W \quad (1)$$

where:

A = NaOH solution required for blank titration (8.1), mL,

B = NaOH solution required for sample titration (8.2), mL,

N = Normality of NaOH solution,

0.04 = G equivalent NaOH per mL of 1 normal solution, and

W = sample used (volume in mL × specific gravity), g.

9.2 *Nonamine Acid Acceptance*—Calculate the nonamine acid acceptance as equivalent NaOH in weight % as follows:

$$\text{Equivalent NaOH, weight } \% \quad (2)$$

$$= \text{total acid acceptance} - \text{amine acid acceptance.}$$

10. Precision and Bias

10.1 The precision limits of this test method have been determined as 0.01 % (equivalent NaOH, weight %).

10.2 The bias of this test method has not been determined.

11. Test Method B

11.1 Determine the weight % of the acid acceptor (typically an epoxide, amines may also contribute acid acceptance) present by a validated method such as gas chromatography, density or refractive index. It will be necessary to calibrate the instrumentation with known standards of the acid acceptor.

11.2 If the acid acceptor is unknown, it can be identified by GC mass spectroscopy.

11.3 Calculate the acid acceptance by the following formula:

$$\text{Equivalent NaOH, weight, } \% = \frac{40\ X \text{ wt } \% \text{ acid acceptor}}{MW} \quad (3)$$

where:

40 = the molecular weight of NaOH, and

MW = the molecular weight of the acid acceptor.

12. Precision and Bias

12.1 A bias of 102 to 105 % of acid acceptance, as compared to determining acid acceptance by Method A is not uncommon as the yield of acid acceptance from the acid acceptor by Method A is commonly 95 to 98 %. This is actually due to a low bias (95 to 98 %) of Method A rather than high reporting by Method B.

12.2 A Relative Standard Deviation of about 0.8 % can be expected when the weight percent acid acceptor is determined by gas chromatography. The precision of the acid acceptance will be the same as the precision of the weight percent acid acceptor.

13. Keywords

13.1 acid acceptance; halogenated organic solvents; solvents; total acid acceptance

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[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.

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