Designation: D1613 – 17 (Reapproved 2023)

Standard Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products[1]

This standard is issued under the fixed designation D1613; 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 total acidity as acetic acid, in concentrations below 0.05 %, in organic compounds and hydrocarbon mixtures used in paint, varnish, and lacquer solvents and diluents. It is known to be applicable to such mixtures as low molecular weight saturated and unsaturated alcohols, ketones, ethers, esters, hydrocarbon diluents, naphtha, and other light distillate petroleum fractions.

1.2 For purposes of determining conformance of an observed value or a calculated value using this test method to relevant specifications, test result(s) 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 the standard. The values given in parentheses are for information only.

1.4 For specific hazard information and guidance consult 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.*

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]

D770 Specification for Isopropyl Alcohol

D1193 Specification for Reagent Water

D4806 Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel

D7795 Test Method for Acidity in Ethanol and Ethanol Blends by Titration

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 mixed with either an equal volume of water or an equal volume of alcohol, and titrated with aqueous sodium hydroxide solution to the phenolphthalein end point.

4. Significance and Use

4.1 This test method is useful for determining low levels of acidity, below 0.05 %, in organic compounds and hydrocarbon mixtures. The total acidity is calculated as acetic acid or milligrams of sodium hydroxide per gram of sample.

4.2 Acidity may be present as a result of contamination, decomposition during storage or distribution, or manufacture. This test method may be used in assessing compliance with a specification.

5. Apparatus

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

5.2 *Erlenmeyer Flask,* 250 mL capacity.

[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.

6. Purity of Reagents

6.1 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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D1193.

7. Reagents

7.1 *Alcohols,* refined, ethyl or isopropyl.

NOTE 1—Isopropyl alcohol (99 % grade) conforming to Specification D770, or 190 proof ethyl alcohol conforming to formula No. 3A of the U.S. Bureau of Alcohol, Tobacco and Firearms is suitable for use as the solvent. The use of methyl alcohol is not recommended.

7.2 *Phenolphthalein Indicator Solution (10 g/L)*—Dissolve 1 g of phenolphthalein in ethyl or isopropyl alcohol (see Note 1) and dilute to 100 mL with the alcohol.

7.3 *Sodium Hydroxide, Standard Solution (0.05 N)*—Prepare and standardize a 0.05 *N* sodium hydroxide (NaOH) solution (Note 2) in accordance with the Preparation and Standardization of Solutions, Precision and Bias, Preparation of 50 % of NaOH Solution and of Standard Solutions, and Standardization sections of Practice E200.

NOTE 2—Alternatively, KOH solution may be used.

8. Procedure

8.1 Measure into a 250 mL Erlenmeyer flask 50 mL of water, if the sample is completely water-soluble, or 50 mL of alcohol, if the sample is not completely water-soluble.

8.2 Add 0.5 mL of phenolphthalein indicator solution. Titrate the water or alcohol with 0.05 *N* NaOH solution to the first perceptible pink color.

8.3 Pipet 50 mL of the sample into the flask. Titrate with the 0.05 *N* NaOH solution to the same first perceptible pink color originally obtained.

8.3.1 If the sample is fuel ethanol or denatured fuel ethanol, as defined in Specification D4806, purge the sample for 2 min with 400 mL/mn of nitrogen prior to pipetting (see Test Method D7795 for additional information on purging the sample).

NOTE 3—It is well known that carbon dioxide is highly soluble in ethanol solutions. Carbon dioxide can be dissolved into the ethanol solution during production (especially fermentation processes), during transportation or during laboratory analysis. The dissolved carbon dioxide is a known interference for this test method creating incorrect elevated acidity values. If the dissolved carbon dioxide is not removed from a sample, erroneously high results can be experienced which can exceed the specification limit. It is recommended that Test Method D7795 be used for the determination of acidity in fuel ethanol and denatured fuel ethanol; furthermore, as is codified in Specification D4806, Test Method D7795 should be considered the referee method:

Section 8.4.1 from Specification D4806: "Dissolved carbon dioxide is a known interference and can cause a false high reading when using Test Method D1613. In the absence of dissolved CO_2, Test Method D1613 is an acceptable method. If a sample is known to have dissolved CO_2 or if dissolved CO_2 can be present, Test Method D7795 is the preferred method. In cases of differing results between the two test methods, Test Method D7795 shall be the referee method."

9. Calculations

9.1 Calculate the acidity of the sample as follows:

$$\text{Acidity as acetic acid, weight \%} = (VN \times 0.12)/D \quad (1)$$

or,

$$\text{Acidity as mg KOH per g of sample} = (VN \times 1.12)/D \quad (2)$$

where:

V = NaOH solution required for titration of the sample, mL,
N = normality of the NaOH solution, and
D = density of specimen in g/mL.

10. Report

10.1 Report the percent of acetic acid to the nearest 0.0001 %. Duplicate runs that agree within 0.0005 %, absolute, are acceptable for averaging (95 % confidence level).

11. Precision and Bias[4]

11.1 *Precision:*

11.1.1 The following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1.1 *Repeatability*—The normal range between two results, each the mean of duplicate determinations, obtained by the same analyst on different days, is estimated to be 0.0003 %, absolute. Two such values should be considered suspect if they differ by more than 0.0008 %, absolute.

11.1.1.2 *Reproducibility*—The normal range between two results, each the mean of duplicate determinations obtained by analysts in different laboratories, is estimated to be 0.0005 %, absolute. Two such values should be considered suspect if they differ by more than 0.0014 %, absolute.

NOTE 4—The above precision estimates are based on an interlaboratory study on two samples each of *n*-butyl acetate, *n*-butyl alcohol, and methyl ethyl ketone containing 0.0058, 0.0112, 0.0007, 0.0046, 0.0026, and 0.0067 % acetic acid, respectively. Each of four laboratories analyzed all six samples, with two analysts in each laboratory performing duplicate determinations using both 99 % isopropyl alcohol and formula 3A ethanol as solvents, and repeating on a second day, for a total of 384 determinations.

11.2 *Bias*—The bias of this test method has not been determined because there is no available material with an accepted reference value.

[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.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1041. Contact ASTM Customer Service at service@astm.org.