Designation: D 2086 – 03

Standard Test Method for Acidity in Vinyl Acetate and Acetaldehyde[1]

This standard is issued under the fixed designation D 2086; 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.

1. Scope*

1.1 This test method covers the determination of total acidity as acetic acid in refined vinyl acetate and acetaldehyde.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 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 E 29.

1.4 For hazard information and guidance, see the supplier's Material 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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 8.

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 mixed with either an equal volume of chilled water or an equal volume of ethyl alcohol and titrated at reduced temperature with aqueous sodium hydroxide solution to a phenolphthalein end point.

4. Significance and Use

4.1 This test method provides a measurement of total acidity in vinyl acetate and acetaldehyde. The results of these measurements can be used for specification acceptance.

5. Interferences

5.1 Any material or contaminant that will react with NaOH under the test conditions will affect the results.

5.2 Vinyl acetate will decompose on storage, typically by way of hydrolysis, to form acetic acid.

5.3 Acetaldehyde will react with oxygen, either dissolved or in a storage container, to form acetic acid.

5.4 Various acids or other acidic materials may be present. Common practice, including the method used here, calculates these as acetic acid. The actual weight percent of acidic materials may be different.

6. Apparatus

6.1 *Buret*, 10-mL, graduated in 0.05-mL subdivisions.

6.2 *Erlenmeyer Flask*, 250-mL capacity.

6.3 *Graduated Cylinder*, 50 or 100-mL capacity.

6.4 *Cold Bath*, maintained at 0°C or below.

7. Reagents

7.1 *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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

7.3 *Ethyl Alcohol (Ethanol)*, 95 volume %, minimum.

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

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

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[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.

***A Summary of Changes section appears at the end of this standard.**

NOTE 1—Denatured ethyl alcohol conforming to Formula No. 3A of the U.S. Bureau of Alcohol, Tobacco and Firearms is suitable for use as a solvent.

7.4 *Phenolphthalein Indicator Solution* (10 g/L)—Dissolve 1 g of phenolphthalein in 100 mL of methanol, ethanol, or isopropanol.

7.5 *Sodium Hydroxide, Standard Solution* (0.05 *N*)—Prepare and standardize a 0.05 *N* sodium hydroxide (NaOH) solution (Note 2) in accordance with the Sodium Hydroxide Solution sections of Practice E 200.

NOTE 2—Alternatively, potassium hydroxide (KOH) solution may be used.

7.6 *Bromothymol Blue Indicator (10g/L)*—Dissolve 1 g of bromothymol blue in 100 mL of methanol, ethanol, or isopropanol.

7.7 *Sodium Hydroxide, Standard Solution (0.02 N)*—Prepare and standardize a 0.02 *N* sodium hydroxide (NaOH) solution (Note 3) in accordance with the Sodium Hydroxide Solution sections of Practice E 200.

NOTE 3—Alternatively, potassium hydroxide (KOH) solution may be used. If the titration solution becomes cloudy due to water saturation during titrations, 0.02 *N* alcoholic KOH may be used as the titrant.

8. Hazards

8.1 Vinyl acetate and acetaldehyde are flammable and hazardous as their vapors form explosive mixtures with air.

8.2 Acetaldehyde boils at 21°C; therefore, store in pressure containers or refrigerate if kept in glass containers. Wear safety goggles or a full face shield when handling acetaldehyde.

9. Procedure for Vinyl Acetate (**Warning**—see Section 8)

9.1 If a solvent is to be utilized, add 50 mL of ethanol or anhydrous methanol. Alternatively, the vinyl acetate may be titrated neat.

9.2 If a solvent is utilized, add 0.5 mL bromothymol blue indicator to the solvent and titrate with 0.02 *N* NaOH solution to the first blue color.

9.3 Add 50 mL of the specimen from a graduated cylinder and cool the solution to approximately 0°C in a cold bath (alternatively, chill in refrigerator for 15 min. If a solvent is not used and the indicator was not added in 9.2, add 0.5 mL of bromothymol blue indicator. The solution may be purged to remove any absorbed carbon dioxide.

9.4 Titrate with the 0.02 *N* NaOH solution to the blue color.

10. Procedure for Acetaldehyde (**Warning**—see Section 8)

10.1 Measure into a 250-mL Erlenmeyer flask 50 mL of water and add sufficient crushed ice (prepared from reagent water) so that some ice will remain at the end of the determination.

NOTE 4—Titration at low temperature avoids interference and is required because of the low boiling point of acetaldehyde.

10.2 Add 0.5 mL of phenolphthalein indicator solution and titrate with 0.05 *N* NaOH solution to the first perceptible pink color.

10.3 By means of a graduated cylinder add 50 mL of the acetaldehyde specimen that previously has been chilled to 0 to 5°C.

10.4 Titrate *immediately* with the 0.05 *N* NaOH solution to the same first perceptible pink color originally obtained in 10.2.

11. Calculation

11.1 Calculate the acidity *A* of the specimen as follows:

11.1.1 Acidity as weight % acetic acid:

$$A = (VN \times 0.060 \times 100)/50\,D = (VN \times 0.12)/D \quad (1)$$

or

11.1.2 Acidity as milligrams KOH per gram of specimen:

$$= (VN \times 0.056 \times 1000)/50\,D = (VN \times 1.12)/D \quad (2)$$

where:

V	=	volume of NaOH solution required for titration of the specimen, mL,
N	=	normality of the NaOH solution,
D	=	density of specimen in g/mL,
0.060	=	milliequivalent weight of acetic acid, and
0.056	=	milliequivalent weight of KOH.

12. Report

12.1 Report the following information:

12.1.1 The percent of acetic acid to the nearest 0.0001 %.

12.1.2 *Vinyl Acetate*—Acceptable duplicate determinations for averaging have not yet been determined.

12.1.3 *Acetaldehyde*—Duplicate determinations that agree within 0.010 %, absolute, are acceptable for averaging (95 % confidence level).

13. Precision and Bias

13.1 The following criteria should be used for determining the acceptability of results at the 95 % confidence level.

13.2 *Vinyl Acetate*:

13.2.1 *Repeatability*—The repeatability of this test method has not yet been determined.

13.2.2 *Reproducibility*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated at 0.0002 wt. % absolute with 3 df. The 95 % limit for differences between two such averages is estimated at 0.0006 wt. %.

NOTE 5—The statistical data was determined with only two laboratory participants and will be utilized only until a complete round robin study can be completed.

13.3 *Acetaldehyde*:

13.3.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same analyst should be considered suspect if they differ by more than 0.014 % absolute.

13.3.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by analysts in different laboratories should be considered suspect if they differ by more than 0.035 %, absolute.

NOTE 6—The above precision estimates are based on an interlaboratory study on samples of acetaldehyde containing 0.034 and 0.146 % acetic acid. Each sample was analyzed in duplicate by two analysts in each of five different laboratories on two different days.

13.4 *Bias*—Bias has not been determined for this test method because an appropriate standard is not available (see Section 5).

14. Keywords

14.1 acetaldehyde; acidity; vinyl acetate

SUMMARY OF CHANGES

Committee D01.35 has identified the location of selected changes to this standard since the last issue (D 2086 - 02) that may impact the use of this standard.

(1) Changed the definition of "D" gravity in equations 1 and 2 in 11.1.1 and 11.1.2 from specific gravity to "density in g/mL."