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Standard Test Method for Acetaldehyde Content of Vinyl Acetate[1]

This standard is issued under the fixed designation D 2191; 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 trace quantities of acetaldehyde, in the range from 0.00 to 0.05 %, contained in 99 % grade vinyl acetate.

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.* For specific hazard statements see Section 8.

1.3 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

3. Summary of Test Method

3.1 The acetaldehyde present in the specimen is reacted with a measured excess of sodium bisulfite. The amount of sodium bisulfite consumed, determined by titrating the excess with a standard iodine solution, is a measure of the acetaldehyde present in the vinyl acetate.

4. Significance and Use

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

5. Interference

5.1 Ketones and other aldehydes, if present, cause a positive interference.

6. Apparatus

6.1 *Buret*, 50-mL capacity, graduated in 0.1-mL subdivisions, with a funnel or flared top and a ground-glass stopcock.

6.2 *Erlenmeyer Flask*, 500-mL capacity, glass-stoppered.

6.3 *Pipet*, 50-mL capacity.

6.4 *Pipet*, 100-mL capacity.

7. Reagents

7.1 *Purity of Reagents*—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 that 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 *Iodine, Standard Solution* (0.1 *N*)—Dissolve 35.0 g of potassium iodide (KI) and 13.0 g of resublimed iodine in water, and dilute to 1 L with water. Store this solution in a dark bottle and standardize each day, as required, against a standard 0.1 *N* sodium thiosulfate ($Na_2S_2O_3$) solution. (See standardization procedure, 9.3 and 9.4.)

7.4 *Potassium Iodate* (KIO_3), primary standard.

7.5 *Sodium Bisulfite Solution* (0.44 %)—Dissolve 4.4 g of sodium metabisulfite ($Na_2S_2O_5$) in 1 L of water. This solution should be prepared fresh daily or just before using.

7.6 *Sodium Thiosulfate, Standard Solution* (0.1 *N*)— Weigh to 0.1 g 24.8 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) crystals and dissolve in 500 mL of water. Dilute this solution to 1 L with water. Add 0.5 mL of chloroform per litre of solution as a preservative, and store in a clean dark bottle. This solution should be standardized weekly, as required, against potassium iodate (KIO_3). (See standardization procedure, 9.1 and 9.2.)

7.7 *Starch Indicator*—Make a paste of 6 g of powdered soluble starch in water, and dilute to 1 L with water, stirring to produce a suspension. While stirring, add 20 g of potassium hydroxide (KOH) pellets, and continue stirring until the KOH is dissolved. Let stand for 2 h and add 27.5 mL of hydrochloric acid (HCl). Adjust the mixture to a pH of 6.0 ± 0.1 by adding small increments of HCl or KOH as required. Add 6 mL of glacial acetic acid as a preservative.

[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] *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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