Designation: D 1979 – 97

Standard Test Method for Free Formaldehyde Content of Amino Resins[1]

This standard is issued under the fixed designation D 1979; 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 free formaldehyde in amino resins and their aqueous and non-aqueous solutions. Amino resin-free formaldehyde levels from about 0.02 to 5.0 % can be determined by this test method. The applicability of this test method to other matrices is unknown.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids[3]

3. Summary of Test Method

3.1 Specimens are mixed with borate buffer solution and ice water, then kept cool in an ice bath. Excess sodium sulfite is added to consume free formaldehyde as follows:

$$CH_2O + Na_2SO_3 + H_2O \rightarrow HOCH_2SO_3Na + NaOH \quad (1)$$

3.2 The excess sodium sulfite is removed by titration with iodine using starch as the indicator as follows:

$$Na_2SO_3 + I_2 + H_2O \rightarrow Na_2SO_4 + 2\,HI \quad (2)$$

3.3 The sodium sulfite-formaldehyde complex is then decomposed with sodium carbonate to quantitatively regenerate sodium sulfite and formaldehyde as follows:

$$HOCH_2SO_3Na + Na_2CO_3 \rightarrow CH_2O + Na_2SO_3 + NaHCO_3 \quad (3)$$

3.4 The liberated sodium sulfite is titrated with iodine (Eq 2) and free formaldehyde is calculated from this second iodine titration.

4. Significance and Use

4.1 The amount of free formaldehyde in amino resins may be of concern to both producer and user, as its presence in air above threshold amounts may produce objectionable odors and irritant effects. This test method can be useful for evaluating suppliers' products and for quality control.

5. Apparatus

5.1 *Analytical Balance*, ±0.1 mg.

5.2 *Magnetic Stirrer and Stir Bar*—A "heavy duty" magnetic stirrer is necessary.

5.3 *Buret*, 50 mL manual or electronic. Manual burets should be of the type designed to minimize the exposure of reagent to air. Electronic burets are preferred.

5.4 *Beakers*, glass, 100 mL, 600 mL, and 1500 mL.

5.5 *Stopwatch or Timer*.

5.6 *Graduated Cylinders*, glass, 50 mL and 250 mL.

5.7 *Glass Pipet*, 2 mL.

5.8 *Thermometer*, subdivision 1°C.

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 conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficient 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 as defined by Type II of Specification D 1193.

6.3 *Acetic Acid Solution (1.0 M)*—Dissolve 60 mL of glacial acetic acid (CH_3CO_2H) in water and dilute to 1 L.

6.4 *Boric Acid Buffer Solution*—Prepare a sodium hydroxide solution (1.0 *M*) by dissolving 40 g of sodium hydroxide

[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

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

[3] *Annual Book of ASTM Standards*, Vol 06.03.

[4] *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.