



Standard Test Method for Iron in Formaldehyde Solutions¹

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1. Scope

1.1 This test method covers the determination of the total iron content of formaldehyde solutions.

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.*

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 Water²

3. Summary of Test Method

3.1 The specimen is evaporated and ashed; the iron, reduced to the divalent state by the addition of hydroxylamine hydrochloride, is reacted with *o*-phenanthroline to develop a color that is measured at 510 nm.

4. Significance and Use

4.1 This test method provides a measurement of iron content of formaldehyde solutions. The results of these measurements can be used for specification acceptance.

5. Apparatus

5.1 *Spectrophotometer*, capable of measuring light absorption at 510 nm.

5.2 *Absorption Cells*, minimum light path, 10 mm.

5.3 *Evaporating Dishes*, 90-mm diameter, high-silica glass.

6. Reagents and Materials

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,

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 as defined by Type IV of Specification D 1193.

6.3 *Ammonium Acetate Solution* (100 g/L)—Dissolve 100 g of ammonium acetate ($\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$) in 100 mL of water. Add 200 mL of acetic acid (CH_3COOH), dilute to 1 L with water, and mix.

6.4 *Ammonium Hydroxide* (1 + 1)—Mix equal volumes of concentrated ammonium hydroxide (NH_4OH , sp gr 0.90) and water.

6.5 *Congo Red Paper*.

6.6 *Hydrochloric Acid* (1 + 1)—Mix equal volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) and water.

6.7 *Hydroxylamine Hydrochloride Solution* (100 g/L)—Dissolve 10 g of hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) in water and dilute to 100 mL.

6.8 *Iron, Standard Solution* (1 mL = 0.05 mg Fe)—Dissolve 0.3510 g of ferrous ammonium sulfate ($\text{FeSO}_4\cdot(\text{NH}_4)_2\text{SO}_4\cdot 6\text{H}_2\text{O}$) in 50 mL of water and 20 mL of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84). Dilute with water to 1 L in a volumetric flask and mix.

6.9 *o-Phenanthroline Solution* (1 g/L)—Dissolve 0.1 g of *o*-phenanthroline in 10 mL of iron-free ethyl alcohol⁴ and dilute to 100 mL with water.

7. Calibration

7.1 Prepare a series of standards by adding the reagents described in 9.3 to 0.2, 0.5, 1.0, 2.0, 3.0, 4.0, and 5.0-mL of standard iron solution in 100-mL volumetric flasks, and diluting to volume.

7.2 Make spectrophotometer comparisons in the absorption cells and prepare a calibration curve by plotting the absorbances of the standard iron solutions against the milligrams of iron per 100 mL of solution. This curve must be determined for each instrument and should be checked periodically.

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 D01.35 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," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analytical Standards for Laboratory U.K., Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

⁴ Specially denatured ethyl alcohol conforming to Formula No. 30 of the U. S. Treasury Department, Bureau of Industrial Alcohol has been found satisfactory for this purpose.