



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.

8. Procedure

8.1 Clean a high-silica glass evaporating dish as follows: Add 10 mL of HCl (1 + 1), cover with a watch glass, and digest on a steam bath for about 20 min. Then discard the HCl solution, rinse the dish with water, and dry.

8.2 Weigh 50 g of sample into the cleaned dish and evaporate to dryness on an electric hot plate in a hood. If any organic matter remains, ignite for 5 min over a high-temperature gas burner.

8.3 Add 10 mL of HCl (1 + 1), cover with a watch glass, and digest on the steam bath for 15 min. Transfer quantitatively to a 100-mL volumetric flask. Add the following reagents in order, mixing after the addition of each: 1 mL of $\text{NH}_2\text{OH}\cdot\text{HCl}$ solution, 5 mL of *o*-phenanthroline solution, enough NH_4OH (1 + 1) as required to make the solution just alkaline to Congo red paper (approximate pH range 3.0 to 5.0), and 5 mL of $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ solution. Dilute to the mark with water and mix thoroughly.

8.4 Allow to stand for 5 min, fill an absorption cell, and measure the absorbance at 510 nm with the spectrophotometer. From the calibration curve, read the milligrams of iron present.

8.5 Carry a blank containing no added iron through all steps of the procedure in order to correct for any iron contamination in the reagents. Correct the results obtained in 8.4 accordingly.

9. Calculation

9.1 Calculate the parts per million of iron *I* as follows:

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$$I = (W/S) \times 1000$$

where:

W = weight, of iron found mg, and

S = weight of sample used, g.

10. Report

10.1 Report the mass of iron to the nearest parts per million.

11. Precision and Bias

11.1 *Repeatability*—The difference between two determinations by the same operator is normally about 0.6 ppm. Two such results should be considered suspect if they differ by more than 0.16 ppm.

11.2 *Reproducibility*—The difference between two results obtained by operators in different laboratories is normally about 0.11 ppm. Two such results should be considered suspect if they differ by more than 0.3 ppm.

11.3 *Bias*—Bias cannot be determined for this test method because there is no material having an accepted reference value.

12. Keywords

12.1 formaldehyde solutions; iron