Designation: D2087 – 06 (Reapproved 2012)

Standard Test Method for Iron in Formaldehyde Solutions[1]

1. Scope*

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

1.2 For purposes of determining conformance of an observed 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 E29.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

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 D1193.

6.3 *Ammonium Acetate Solution (100 g/L)*—Dissolve 100 g of ammonium acetate ($NH_4C_2H_3O_2$) 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.

---

[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.

Current edition approved June 1, 2012. Published July 2012. Originally approved in 1962. Last previous edition approved in 2006 as D2087 – 06. DOI: 10.1520/D2087-06R12.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[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.

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

6.7 *Hydroxylamine Hydrochloride Solution (100 g/L)*—Dissolve 10 g of hydroxylamine hydrochloride ($NH_2OH·HCl$) 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 ($FeSO_4·(NH_4)_2SO_4·6H_2O$) 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 alcohol[4] and dilute to 100 mL with water.

7. Calibration

7.1 Prepare a series of standards by adding the reagents described in 6.3 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.

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 $NH_2OH·HCl$ 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 $NH_4C_2H_3O_2$ 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:

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

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

---

[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.

SUMMARY OF CHANGES

Committee D01.35 has identified the location of selected changes to this standard since the last issue (D2087 – 97 (2001)) that may impact the use of this standard.

(1) Added reference to Practice E29 in the Scope section.

(2) Added Practice E29 to list of Referenced Documents.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).