Designation: E1615 – 16

Standard Test Method for Iron in Trace Quantities Using the FerroZine Method[1]

This standard is issued under the fixed designation E1615; 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 iron in the range from 0.01 to 0.2 µg/g using FerroZine[2] reagent solution. The range may be extended through the use of a 5- or 10-cm cell or by suitable dilution of the sample solution.

1.2 This test method is intended to be general for the final steps in the determination of iron and does not include procedures for sample preparation.

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 Review the current Safety Data Sheets (SDS) for detailed information concerning toxicity, first-aid procedures, and safety precautions.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 7.4.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1193 Specification for Reagent Water

E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[4]

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Summary of Test Method

3.1 This test method is based upon a photometric determination of the FerroZine[2] complex with the iron (II) ion.[5,6] The sample is dissolved in a suitable solvent and the iron is reacted with FerroZine reagent solution which will convert the dissolved iron compounds to form a magenta color iron (II) complex. The iron content of the sample solution is determined by measurement of the magenta color at 560 nm using a suitable photometer.

4. Significance and Use

4.1 This test method is suitable for determining trace concentrations of iron in a wide variety of products, provided that appropriate sample preparation has rendered the iron and sample matrix soluble in water or other suitable solvent. Each sample matrix must be investigated for suitability using this test method.

4.2 This test method assumes that the amount of color developed is proportional to the amount of iron in the test solution. The calibration curve is linear over the specified range.

5. Interferences

5.1 Any ion that absorbs light at 560 nm will interfere with the determination. Anionic interferences include oxalate in concentrations over 500 µg/g, cyanide, and nitrate.[5]

5.2 Of copper, cobalt, calcium, magnesium, lead, silver, molybdenum, aluminum, nickel, zinc, arsenic, manganese,

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.04 on Instrumental Analysis.

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[2] FerroZine is a trademark of Hach Chemical Company.

[3] 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.

[4] The last approved version of this historical standard is referenced on www.astm.org.

[5] Stookey, L. L., "FerroZine—A New Spectrophotometric Reagent for Iron," *Analytical Chemistry*, Vol 42, No. 7, June 1970, pp. 779 – 781.

[6] Gibbs, C. R., "Characterization and Application of FerroZine Iron Reagent as a Ferrous Iron Indicator," *Analytical Chemistry* , Vol 48, No. 8, July 1976, pp. 1197–1201.

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

hexavalent chromium, trivalent chromium, divalent cobalt and monovalent copper are the only metals other than iron that form colored species with FerroZine under test conditions. At least 1000 mg/L of the alkali metals and the alkaline earths had no effect on the determination. Many heavy metals will react with FerroZine in competition with iron, but with the excess reagent used in the test there is no effect on the results.[5]

5.3 The pH range of the final solution should be from 4 to 9 to give the best test results.[5,6]

5.4 All glassware used in this test method must be iron-free and scrupulously clean by precleaning with dilute hydrochloric acid and FerroZine reagent solution followed by a water rinse.

6. Apparatus

6.1 *Photometer,* capable of measuring light absorption at 560 nm and holding a 5-cm or 10-cm cell. Check the performance of the photometer at regular intervals according to the guidelines given in Practice E275 and the manufacturer's manual.

6.2 *Absorption Cells,* 5-cm or 10-cm light path.

NOTE 1—A discussion of photometers and photometric practice is given in Practice E60.

7. Reagents

7.1 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.[7] 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II or Type III reagent water conforming to Specification D1193.

7.3 *Iron, Standard Solution,* 1 mL = 1 µg Fe (see Notes 2 and 3)—Dissolve 0.1000 g of iron wire in 10 mL of hydrochloric acid (HCl, 1 + 1) and 1 mL of saturated bromine water (400 mL water + 20 mL bromine). Boil until the excess bromine is removed. Add 200 mL of HCl, cool, and dilute to 1 L in a volumetric flask. Dilute 10 mL of this solution to 1 L.

NOTE 2—The preparation of this reagent is also described in Practice E200.

NOTE 3—As an alternative, the standard iron solution may be prepared by diluting 1.00 mL of commercially available iron standard stock solution (1000 mg iron/L) to 1 L with water.

7.4 *FerroZine Reagent Solution*—Contains FerroZine color reagent [3-(2-pyridyl)-5,6-bis(4-phenylsulfonic acid)-1,2,4-triazine, monosodium salt, monohydrate], buffer, and a reducing agent. (**Warning—**This solution contains thiols as reducing agents. Wear butyl rubber or neoprene gloves when handling the solution and avoid inhalation of the vapors.)

7.4.1 Alternatively, the individual solutions can be prepared as described below.[5]

7.4.1.1 *Reducing Agent*—Hydroxylamine hydrochloride, 10 percent by weight solution in hydrochloric acid: Dissolve 10 g of reagent grade hydroxylamine hydrochloride ($NH_2OH.HCL$) in 30 g of deionized water in a plastic bottle; add 50 mL of reagent grade concentrated hydrochloric acid and mix well. Prepare this solution fresh daily.

7.4.1.2 *Color Reagent*—FerroZine, 0.514 weight percent solution: Dissolve 0.514 g of FerroZine reagent in 100 g of deionized water in a plastic bottle, and mix well. Discard the reagent after seven days.

7.4.1.3 *Buffer Reagent-pH 10.0 Buffer*—Dissolve 200 g of reagent grade ammonium acetate in a minimum of deionized water, add 175 mL of concentrated ammonium hydroxide and dilute to 500 mL in a volumetric flask. Mix well. Check the pH of the buffer to verify that it is pH 10 ± 0.5. If it is not in the required pH range, remake the buffer. Store the buffer in a plastic bottle. Discard after four weeks.

8. Sampling

8.1 Because this is a general test method for the final steps in determining iron, specific procedures for sample preparation are not included (see 4.1 and 4.2).

9. Calibration

9.1 *FerroZine Reagent Solution Method (7.4):*

9.1.1 By means of suitable pipets or a buret, transfer 0 (reagent blank), 2.0, 4.0, 6.0, 8.0, and 10.0 mL, respectively, of the standard iron solution and approximately 20 mL of water to each of six clean, dry, 50-mL, glass-stoppered volumetric flasks. These flasks represent 0, 2.0, 4.0, 6.0, 8.0, and 10.0 µg of iron. Add 2.0 mL of FerroZine reagent solution to each flask, dilute the contents of each flask to volume with water, stopper, and mix well by inverting the flasks several times. Let the solutions stand for a minimum of 5 min but not more than 10 min to develop the magenta color. Measure the absorbance of each calibration standard in accordance with 10.3.

9.2 *Individual Solution Method (7.4.1):*

9.2.1 By means of suitable pipets or a buret, transfer 0 (reagent blank), 2.0, 4.0, 6.0, 8.0, and 10.0 mL, respectively, of the standard iron solution and approximately 40 mL of water to each of six clean, dry, 100-mL, glass-stoppered volumetric flasks. These flasks represent 0, 2.0, 4.0, 6.0, 8.0, and 10.0 µg of iron. Add the individual reagents (reducing reagent, color reagent and buffer reagent) as described in 10.2 and 10.3 to each flask, dilute the contents of each flask to volume with water, stopper, and mix well by inverting the flasks several times. Let the solutions stand for a minimum of 5 min but not more than 20 min to develop the magenta color. Measure the absorbance of each calibration standard in accordance with 10.3.

9.3 Construct a calibration graph by plotting the absorbances against the corresponding micrograms of iron present in the calibration solutions, including the blank. Obtain the best straight line through the points (calibration function) by

[7] *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. (USP), Rockville, MD.