



Designation: E246 – 21

Standard Test Methods for Determination of Iron in Iron Ores and Related Materials by Dichromate Titrimetry¹

This standard is issued under the fixed designation E246; 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 These test methods cover the determination of total iron in iron ores, concentrates, and agglomerates in the concentration range 30 % to 95 % iron.

1.2 The test methods in this standard are contained in the sections indicated as follows:

Test Method A—Iron by the Hydrogen Sulfide Reduction Dichromate Titration Method (30 % to 75 % Fe)

Test Method B—Iron by the Stannous Chloride Reduction Dichromate Titration Method (35 % to 95 % Fe)

Test Method C—Iron by the Silver Reduction Dichromate Titration Method (35 % to 95 % Fe)

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 *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.* Specific hazards statements are given in Section 5 and in special “Warning” paragraphs throughout these test methods.

1.5 *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:*²

D1193 Specification for Reagent Water

¹ These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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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.

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition and Physical Properties

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1028 Test Method for Total Iron in Iron Ores and Related Materials by Dichromate Titrimetry (Withdrawn 2003)³

3. Significance and Use

3.1 The determination of the total iron content is the primary means for establishing the commercial value of iron ores used in international trade.

3.2 These test methods are intended as referee methods for the determination of iron in iron ores. It is assumed that all who use these test methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices must be followed, such as those described in Guide **E882**.

4. Apparatus, Reagents, and Instrumental Practices

4.1 *Apparatus*—Specialized apparatus requirements are listed in the “Apparatus” Section in each test method.

4.2 *Reagents*:

4.2.1 *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

³ The last approved version of this historical standard is referenced on www.astm.org.

such specifications are available.⁴ 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.

4.2.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or Type II of Specification D1193. Type III or Type IV may be used if they effect no measurable change in the blank or sample.

5. Hazards

5.1 For precautions to be observed in the use of certain reagents and equipment in this test method refer to Practices E50.

6. Sampling and Sample Preparation

6.1 Collect and prepare the test sample in accordance with Practice E877.

6.2 The test sample shall be pulverized to pass a No. 100 (150- μ m) sieve in accordance with Test Method E276. To facilitate decomposition some ores, such as specular hematite, require grinding to pass a No. 200 (75- μ m) sieve.

TEST METHOD A—IRON BY THE HYDROGEN SULFIDE REDUCTION DICHROMATE TITRATION METHOD

7. Scope

7.1 This test method covers the determination of total iron in iron ores, concentrates, and agglomerates in the range from 30 % to 75 % by mass.

7.2 *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.*

8. Summary of Test Method

8.1 The sample is dissolved in HCl. The insoluble residue is removed by filtration, ignited, treated for the recovery of iron, and added to the main solution. To this solution containing all of the iron, H_2SO_4 is added and the solution evaporated to fumes to expel chlorides. The salts are dissolved in water, the solution heated to boiling, and the iron reduced by a rapid stream of hydrogen sulfide (H_2S). The precipitated sulfides are filtered and washed with an acid-sulfide wash solution until free of iron. The filtrate is then boiled to expel the H_2S , cooled, and titrated with $\text{K}_2\text{Cr}_2\text{O}_7$ solution, using sodium diphenylamine sulfonate as the indicator.

9. Interferences

9.1 None of the elements normally found in iron ores interfere with this test method. These include vanadium,

copper, and small amounts of molybdenum, which occasionally occur in iron ores.

10. Apparatus

10.1 *Hydrogen Sulfide Generator*— H_2S shall be obtained from a cylinder of the compressed gas or from a Kipp generator. A consistent flow of 1 L/min shall be maintained and the gas passed through a water trap to remove any salts.

10.1.1 **Warning**— H_2S is extremely toxic. All procedures involving its use must be performed in an efficient fume hood. Refer to Hazards section in Practices E50.

10.2 *Crucibles*, platinum, 25-milliliter (mL) capacity.

11. Reagents and Materials

11.1 *Ferrous Ammonium Sulfate Solution* (approximately 0.10 N)—Dissolve 40 g of ferrous ammonium sulfate ($\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$) in H_2SO_4 (1 + 19). Transfer to a 1-L flask and dilute to volume with the same acid. When the sample solution is ready for titration, standardize the $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ solution against the standard $\text{K}_2\text{Cr}_2\text{O}_7$ (0.1000 N), as described in 12.5. Calculate the milliliters of standard $\text{K}_2\text{Cr}_2\text{O}_7$ equivalent to 1 mL of the $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ solution.

11.2 *Potassium Dichromate, Standard Solution* (0.1000 N)—Transfer 4.9031 g of primary standard grade potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$); previously ground in an agate mortar, and dried at 105 °C to 110 °C, to a 1-L volumetric flask. Dissolve in water and dilute to 1 L. If preferred, this solution may be prepared from reagent grade $\text{K}_2\text{Cr}_2\text{O}_7$, by purifying the salt twice by recrystallizing from water, drying at 110 °C, pulverizing in an agate mortar, and drying at 180 °C to constant weight. The titer of this solution shall be confirmed by means of standard sample similar in type and composition to the test sample.

11.3 *Potassium Permanganate Solution* (25 g/L)—Dissolve 25 g of potassium permanganate (KMnO_4) in water and dilute to 1 L.

11.4 *Sodium Diphenylamine Sulfonate Indicator Solution*—Dissolve 0.3 g of sodium diphenylamine sulfonate in 100 mL of water. Store in a dark-colored bottle.

11.5 *Sodium Pyrosulfate* ($\text{Na}_2\text{S}_2\text{O}_7$).

11.6 *Sulfuric Acid-Hydrogen Sulfide Wash Solution*—Add 20 mL of concentrated H_2SO_4 (H_2SO_4 , sp gr 1.84) to 900 mL water, cool, dilute to 1 L, and pass a rapid stream of H_2S through it for at least 10 min.

12. Procedure

12.1 Transfer approximately 0.50 g of the test specimen to a small weighing bottle previously dried at about 105 °C. Dry the bottle and contents for 1 h at 105 °C to 110 °C (Note 1). Cap the bottle and cool to room temperature in a desiccator. Momentarily release the cap to equalize the pressure and weigh the capped bottle and sample to the nearest 0.1 mg. Repeat the drying and weighing until there is no further mass loss. Transfer the test specimen to a 250-mL beaker and reweigh the capped bottle to the nearest 0.1 mg. The difference between the two masses is the mass of the sample taken for analysis.

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 the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.