Designation: E 246 – 95

Standard Test Method for
Iron in Iron Ores and Related Materials by Hydrogen Sulfide Reduction and Dichromate Titration[1]

This standard is issued under the fixed designation E 246; 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 total iron in iron ores, concentrates, and agglomerates in the concentration range from 30 to 75 %.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

E 276 Test Methods for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal Bearing Ores and Related Materials[3]

E 877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials[4]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[4]

3. Summary of Test Method

3.1 The sample is dissolved in hydrochloric acid. The insoluble residue is removed by filtration, ignited, and treated for the recovery of iron, and added to the main solution. To this solution containing all the iron, sulfuric acid 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. 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 hydrogen sulfide, cooled, and titrated with potassium dichromate solution using sodium diphenylamine sulfonate as the indicator.

4. Significance and Use

4.1 This test method is intended to be used for compliance with compositional specifications for iron content. It is assumed that all who use these procedures 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 E 882.

5. Interferences

5.1 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.

6. Apparatus

6.1 *Analytical Balance and Weights*—The balance used to weigh the sample shall be sensitive to 0.05 mg and shall have a precision of 0.1 mg. Analytical weights shall be of precision grade or calibrated against a set of certified standard weights.

6.2 *Buret*—The buret used to titrate the reduced solution shall be of precision grade, calibrated to tenths of a millilitre, and shall be read to 0.05 mL.

6.3 *Hydrogen Sulfide Generator*—Hydrogen sulfide 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.

6.4 *Glassware*—Beakers or flasks used in the analysis of the sample shall be of chemical-resistant glass and free of etched surfaces. Before using, all glassware shall be cleaned in hot, dilute hydrochloric acid and thoroughly rinsed in water.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

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[1] This test method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Slags, Refractories, Cement, and Related Oxide Materials.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 03.05.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

TABLE 1 Precision Data

Sample	Number of Laboratories	Iron Found %	Repeatability		Reproducibility	
			s_r	R_1 (2.8 s_r)	s_R	R_2 (2.8 s_R)
Seine River Ore	9	57.52	0.125	0.35	0.126	0.35
Knob Lake Ore	9	58.45	0.097	0.27	0.136	0.38
NBS 27d (64.96 % Fe)	6	65.01	0.057	0.16	0.085	0.24
Chilean Iron Ore	9	66.11	0.102	0.29	0.172	0.48
Pooled standard deviations[A]			0.101		0.137	

[A] Weighted by degrees of freedom, n for s_r and $(n - 1)$ for s_R where n = number of laboratories.

all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] 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 reagent water conforming to Specification D 1193.

7.3 *Ferrous Ammonium Sulfate Solution* (approximately 0.10 *N*)—Dissolve 40 g of ferrous ammonium sulfate ($FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$) in sulfuric acid (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 $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ solution against the standard potassium dichromate (0.1000 *N*), as described in 9.5. Calculate the millilitres of standard potassium dichromate equivalent to 1 mL of the $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ solution.

7.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

7.5 *Hydrochloric Acid* (1 + 50)—Mix 1 volume of HCl (sp gr 1.19) with 50 volumes of water.

7.6 *Hydrofluoric Acid* (48 %)—Concentrated hydrofluoric acid (HF).

7.7 *Hydrogen Sulfide* (H_2S).

7.8 *Nitric Acid* (85 %)—Concentrated nitric acid (HNO_3).

7.9 *Phosphoric Acid* (sp gr 1.69)—Concentrated phosphoric acid (H_3PO_4).

7.10 *Potassium Dichromate, Standard Solution* (0.1000 *N*)—Transfer 4.9035 g of primary standard grade potassium dichromate ($K_2Cr_2O_7$), previously ground in an agate mortar, and dried at 105 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 $K_2Cr_2O_7$, 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.

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

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

7.13 *Sodium Pyrosulfate* ($Na_2S_2O_7$).

7.14 *Sulfuric Acid* (1 + 1)—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) with 1 volume of water.

7.15 *Sulfuric Acid* (1 + 19)—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) with 19 volumes of water.

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

8. Sampling, Test Specimen, and Test Unit

8.1 The test unit shall be collected and prepared in accordance with Practice E 877.

8.2 The test unit shall be pulverized or ground so that at least 95 % passes a No. 100 (150 µ-m) sieve in accordance with Test Method E 276.

NOTE 1—Some ores, such as specular hematities, may require finer grinding to pass a No. 200 (75-µm) sieve.

9. Procedure

9.1 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 to 110°C (Note 2). 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 weight 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 weights is the weight of the sample taken for analysis.

NOTE 2—Most ores yield their hygroscopic moisture at this temperature. If a drying temperature other than that specified is required, this shall be determined by mutual agreement between manufacturer and purchaser.

9.2 *Decomposition of the Sample*—Moisten the sample with a few millilitres of water and add 25 mL of HCl. Cover the beaker and heat, maintaining a temperature below boiling until most of the dark particles are dissolved and no further attack is apparent. Add 5 mL of HNO_3 and digest for another 15 min. Remove from the source of heat, wash the sides and cover of the beaker, and dilute to 50 mL with warm water. Filter the insoluble residue on a fine-texture paper. Wash the residue with warm HCl (1 + 50) until the yellow color of ferric chloride is no longer observed and then with warm water six to eight

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[5] *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.