



Designation: E 246 – 95

Standard Test Method for Iron in Iron Ores and Related Materials by Hydrogen Sulfide Reduction and Dichromate Titration¹

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 Water²

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 Materials³

E 877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials⁴

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory⁴

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 dichro-

mate 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

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.

Current edition approved April 15, 1995. Published June 1995. Originally published as E 246 – 64 T. Last previous edition E 246 – 68 (1989).

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.