



Standard Test Method for Determination of Iron in Manganese Ores by Hydrogen Sulfide Reduction-Dichromate Titration¹

This standard is issued under the fixed designation E 316; 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 manganese ore in the range from 2 to 20 %.

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:

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials²

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory³

3. Terminology

3.1 **Definitions**—For definitions of terms used in this test method, refer to Terminology E 135.

4. Summary of Test Method

4.1 The sample is decomposed by treatment with hydrochloric, hydrofluoric, and sulfuric acids. Any residue is treated for the recovery of insoluble iron. The iron is reduced with hydrogen sulfide, the insoluble sulfides are removed by filtration, and the excess hydrogen sulfide is expelled by boiling. After cooling, the reduced iron is titrated with a standard potassium dichromate solution using sodium diphenylamine sulfonate as the indicator.

5. Significance and Use

5.1 This test method is intended to be used for compliance with compositional specifications for iron content in manga-

nese ores. 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.

6. Interferences

6.1 None of the elements normally found in manganese ore interfere with this test method.

7. Reagents and Materials

7.1 **Purity and Concentration of Reagents**—The purity and concentration of the common chemical reagents used shall conform to Practices E 50. Special apparatus and reagents required are located in separate sections preceding the procedure.

7.2 **Ferrous Ammonium Sulfate Solution** (approx. 0.10 N)—Dissolve 40 g of ferrous ammonium sulfate ($\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$) 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 $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ solution against the standard potassium dichromate solution (0.1000 N) as described in 11.1. Calculate the millilitres of standard potassium dichromate solution equivalent to 1 mL of the $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ solution.

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

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

7.5 **Hydrogen Sulfide** (H_2S).

7.6 **Hydrogen Sulfide Wash Solution**.

7.6.1 Saturate H_2SO_4 (1+99) with hydrogen sulfide (H_2S). Prepare fresh as needed.

7.7 **Nitric Acid** (sp gr 1.42)—Concentrated nitric acid (HNO_3).

7.8 **Phosphoric Acid** (85 %)—Concentrated phosphoric acid (H_3PO_4).

7.9 **Potassium Dichromate, Standard Solution** (0.1000 N)

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

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² *Annual Book of ASTM Standards*, Vol 03.05.

³ *Annual Book of ASTM Standards*, Vol 03.06.