



Designation: **E316–11** E316 – 17

Standard Test Method for Determination of Iron in Manganese Ores by Hydrogen Sulfide Reduction-Dichromate Titrimetry¹

This standard is issued under the fixed designation E316; 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 %.

NOTE 1—As used in this test method (except as related to the term *relative standard deviation*), “percent” or “%” refers to mass fraction (wt/wt) of the form $\frac{g}{100g}$ or $\frac{1g}{100g}$.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

1.4 *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

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

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

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

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

4. Summary of Test Method

4.1 The sample is decomposed by treatment with hydrochloric, hydrofluoric, HCl, HF and sulfuric H₂SO₄ 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 manganese 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 E882.

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 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.

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 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 such specifications are available.³ 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 Type I or II of Specification **D1193**. Type III or IV may be used if they effect no measurable change in the blank or sample.

7.3 *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 H_2SO_4 acid (1 + 19). Transfer to a 1-L flask and dilute to volume with sulfuric H_2SO_4 acid (1 + 19). 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) (7-77.6) 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.

7.4 *Hydrogen Sulfide*—(H_2S).

7.5 *Hydrogen Sulfide Wash Solution*.

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

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

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

7.6.1 Dissolve 4.9035 g of Primary Standard Grade potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

7.7 *Sodium Diphenylamine Sulfonate Indicator Solution* (2 g/L).

7.7.1 Dissolve 0.20 g of sodium diphenylamine sulfonate in 100 mL of water. Store in a dark-colored bottle.

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

8. Hazards

8.1 For precautions to be observed in this method, refer to Practices **E50**.

8.1.1 **Warning**—Caution should be used for HF in **10.2**.

8.1.2 **Warning**—Caution should be used for H_2S in **10.5**.

9. Sampling and Sample Preparation

9.1 The test unit shall be collected and prepared to maintain the representative iron content in the lot.

9.2 The laboratory sample shall be pulverized to pass a No. 100 (150- μm) 150- μm (No. 100) sieve.

9.3 Weigh approximately (within ± 25 mg) an amount of test sample specified as follows:

Content of Iron, %	Weight of Sample, g
Content of Iron, %	Mass of Sample, g
2 to 10	2.0
10 to 20	1.0

10. Procedure

10.1 Transfer the test sample to a small, dry weighing bottle and place into a drying oven. After drying at 120 °C for 1 h, cap the bottle and cool to room temperature in a desiccator. Momentarily release the cap to equalize pressure and weigh the capped bottle to the nearest 0.1 mg. Repeat the drying and weighing until there is no further weight/mass loss. Transfer the test sample to a 400-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.

10.2 Moisten the sample with a few millilitres of water, add 15 mL of HCl, cover and heat gently until no further attack is apparent. Add 5 mL of HNO_3 and warm until the action subsides. Add 10 drops to 15 drops of HF and 15 mL of H_2SO_4 , and heat to copious fumes of SO_3 (do not prolong fuming). Cool, rinse the sides of the beaker with a few millilitres of water, and again heat to fumes of SO_3 . Cool, add 100 mL of water, and heat to boiling.

³ *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 *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole Dorset, U. K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.