



Designation: E 1081 – 95a

Standard Test Method for Determination of Total Iron in Iron Ores and Related Materials by Silver Reduction-Dichromate Titration¹

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1. Scope

1.1 This test method covers the determination of total iron in ores, concentrates, and agglomerates in the concentration range from 35 to 95 % iron, and is free from vanadium interference.

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.*

1.3 Use of safety goggles is required for fusion with sodium peroxide.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals³

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

E 1028 Test Method for Total Iron in Iron Ores by the Dichromate Titrimetric Method⁴

3. Summary of Test Method

3.1 *Acid Decomposition*—The test sample is dissolved in hydrochloric acid. The insoluble residue is removed by filtration, ignited, treated for the recovery of iron, and added to the main solution.

3.2 *Decomposition by Fusion*—The test sample is fused with sodium peroxide or sintered with sodium peroxide at 400°C, and fused over a burner. The melt is leached with water and acidified with hydrochloric acid.

3.3 *Reduction of Iron*—The test sample solution is passed through a silver-reductor. After addition of phosphoric acid-

sulphuric acid mixture and diphenylamine-sulphonate indicator, the total iron, is determined by titration with a standard solution of potassium dichromate.

4. Significance and Use

4.1 This method for the analysis of iron ores concentrates and agglomerates is primarily intended as a referee method to test for compliance with compositional specifications. It is assumed that all who use this test method 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.

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

5. Interferences

5.1 This test method is written for iron ores containing less than 0.1 % of copper. Other elements, particularly vanadium, normally found in iron ores do not interfere with this test method.

6. Apparatus

6.1 Silver Reductor.

6.1.1 *Preparation of Silver Reductor*—Use a glass column (2 cm in diameter and 25 cm in length) fitted with a stopcock and a reservoir cup (about 100 mL in capacity, 4 cm in diameter, and 9 cm in length).

6.1.1.1 Place glass wool *very lightly* above the stopper. Fill column with HCl (1+11). Place silver powder into a 150-mL beaker. Add HCl (1+11) and transfer the silver powder into the column using HCl (1+11), avoiding the entrapment of air. Pass about 100 mL of HCl (1+11) through the column.

NOTE 1—Care must be taken not to let the column dry. Always maintain about 1 mL of HCl (1+11) above the silver powder.

NOTE 2—The height of silver in the column is about 17 cm and is adequate for about 18 samples prior to regeneration. The flow rate is from 35 to 40 mL/min. Alternatively silver can be prepared by reducing silver nitrate with zinc as follows: Dissolve 50 g of AgNO₃ (6.12) in 400 mL of water in a 600-mL beaker. Add 10 mL of HNO₃ (6.8). Place two zinc metal rods, 15 cm in length, crosswise and leave 4 h or overnight. Wash the precipitated silver thoroughly by decantation using H₂SO₄ (1+99). A glass

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, Concentrates, and Related Metallurgical 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.

column (2 cm in diameter and 15 cm in length) fitted with a stopcock and reservoir cup (about 100 mL in capacity, 4 cm in diameter, and 9 cm in length) is used. Pack glass wool lightly above the stopper. Transfer the washed, precipitated silver into the column using $\text{H}_2\text{SO}_4(1+99)$ and avoiding any trapped air. Wash the column with $\text{HCl}(1+99)$ several times (150 mL are sufficient) at a flow rate of 30 to 35 mL/min. (Length of the silver column is about 9 cm.) The silver reductor is now ready. Always maintain about 1 mL of $\text{HCl}(1+11)$ above the column.

6.1.2 Regeneration of Silver Reductor—With the passage of iron (III) the silver in the reductor darkens at the very top, forming a greyish ring which extends down. When this ring extends down to about 10 cm, the column should be regenerated as follows: Drain the solution (leaving about 1 cm on the top and wash with 150 mL of $\text{H}_2\text{SO}_4(1+99)$). Finally keep the level of $\text{H}_2\text{SO}_4(1+99)$ in the column about 1 cm above the silver. Gently rest two zinc rods (6.20) in contact with the silver in the column. Leave overnight. The dark color changes to silvery white indicating complete regeneration to metallic silver.

NOTE 3—Passing 50 mL of $\text{H}_2\text{SO}_4(1+99)$ through the column accelerates the regeneration. Then wash the column several times by passing $\text{HCl}(1+11)$. The column is ready for re-use. The regeneration can also be done by emptying the contents into a beaker, placing zinc-rods and repacking as in 7.4.1.

NOTE 4—If the flow is slow, remove the silver and the glass wool from the column and repack as in 6.1.1. Ensure that the new glass wool is placed very lightly for restoring the flow rate at 30 to 35 mL/min.

6.2 Weighing Spatula of a nonmagnetic material or demagnetized stainless steel.

6.3 Zirconium (Metal) Crucibles, 50 mL capacity.

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.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficient 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 as defined by Type I of Specification D 1193.

7.3 Ammonium Hydroxide (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

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

7.5 Hydrofluoric Acid (sp gr 1.15)—Concentrated hydrofluoric acid (HF).

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

7.7 Phosphoric Acid (sp gr 1.57)—Concentrated phosphoric acid (H_3PO_4).

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.

7.8 Potassium Dichromate, Standard Solution (0.1 N)—Pulverize about 6 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) reagent in an agate mortar, dry in an air-bath at 140°C for 3 to 4 h, and cool to room temperature in a desiccator. Dissolve 4.9040 g of the dry reagent in water and dilute the solution with water to exactly 1 L in a volumetric flask.

7.9 Potassium Pyrosulfate ($\text{K}_2\text{S}_2\text{O}_7$) *Fine Powder*.

7.10 Silver Nitrate (AgNO_3).

7.11 Silver Powder—Johnson Matthey 40 to 60 mesh is suitable.

7.12 Sodium Diphenylaminesulfonate Solution (2 g/L)—Dissolve 0.2 g of the reagent ($\text{C}_6\text{H}_5\text{NC}_6\text{H}_4\cdot\text{SO}_3\text{Na}$) in water and dilute to 100 mL. Store the solution in a brown glass bottle.

7.13 Sodium Peroxide (Na_2O_2) dry powder.

7.14 Sulfuric Acid (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

7.15 Sulfuric Acid Phosphoric Acid Mixture—Pour 150 mL of phosphoric acid (H_3PO_4) into about 400 mL of water. While stirring, add 150 mL of sulfuric acid (H_2SO_4). Cool in a water bath and dilute with water to 1 L.

7.16 Zinc Metal Rods, about 8 mm in diameter and about 150 mm in length.

8. Sampling and Sample Preparation

8.1 Collect and prepare gross samples in accordance with Practice E 877.

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

NOTE 5—To facilitate decomposition, some ores, such as specular hematite, require grinding to pass a No. 200 (75- μm) sieve.

9. Procedure

NOTE 6—If the procedure is based on acid decomposition, use steps in 9.1. If the procedure is based on fusion, use steps in 9.2.

9.1 Acid Decomposition:

9.1.1 Weigh approximately 0.3 g of prepared sample into a small weighing bottle previously dried at about 105°C . Dry the bottle and contents for 1 h at 105 to 110°C . 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 sample 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 test sample taken for analysis.

9.1.2 Add 20 mL of HCl , cover the beaker with a watch glass, and heat the solution gently without boiling, to decompose the ore. Wash the watch glass with a jet of water and dilute to 50 mL with warm water. Filter the insoluble residue on a close texture paper. Wash the residue with warm $\text{HCl}(1+50)$, until the yellow color of iron (III) chloride is no longer observed. Then wash with warm water six to eight times. Collect the filtrate and washings in a 400-mL beaker.

9.1.3 Place the filter paper and residue in a platinum crucible, dry, and ignite at 750 to 800°C . Allow the crucible to cool, moisten the residue with $\text{H}_2\text{SO}_4(1+1)$, add about 5 mL of $\text{HF}(7.5)$, and heat gently to remove silica and sulfuric acid. Add to the cold crucible 2 g of $\text{K}_2\text{S}_2\text{O}_7$, heat gently at first then strongly until a clear melt is obtained. Cool, place the crucible