



Designation: E 1028 – 98

Standard Test Method for Total Iron in Iron Ores and Related Materials by Dichromate Titrimetry¹

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1. Scope

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

1.2 *This standard does not purport to address all of the safety problems, 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 877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials³

3. Summary of Test Method

3.1 *Acid Decomposition*—The 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 sample is fused with a mixture of sodium carbonate and 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. For samples containing more than 0.1 % vanadium or 0.1 % molybdenum, or both, the melt is leached with water and filtered. The insoluble iron hydroxide is then dissolved in hydrochloric acid.

3.3 *Reduction of the Iron*—Most of the iron is reduced in a hot 6M hydrochloric acid medium with stannous chloride, followed by the addition of a slight excess of titanium (III) solution. The excess Ti (III) is oxidized in the hot solution with perchloric acid. The solution is rapidly cooled and the reduced

iron is titrated with a standard dichromate solution using sodium diphenylamine sulfonate as indicator.

4. Significance and Use

4.1 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.

4.2 This test method is intended to be a referee method for the determination of iron in iron ores. It is assumed that the user of this procedure 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.

5. Interferences

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

6. Apparatus

6.1 *Zirconium*, vitreous carbon, or sintered alumina crucibles, 25 to 30-mL capacity.

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

7. Reagents

7.1 *Purity and Concentration of Reagents*—The purity and concentration of all reagents used shall conform to Practices E 50.

7.2 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

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

7.4 *Hydrochloric Acid* (1 + 1)—Mix 1 volume of concentrated hydrochloric acid (HCl) with 1 volume of water.

7.5 *Hydrochloric Acid* (1 + 2)—Mix 1 volume of concentrated hydrochloric acid (HCl) with 2 volumes of water.

7.6 *Hydrochloric Acid* (1 + 10)—Mix 1 volume of concentrated hydrochloric acid (HCl) with 10 volumes of water.

7.7 *Hydrochloric Acid* (1 + 50)—Mix 1 volume of concentrated hydrochloric acid (HCl) with 50 volumes of water.

7.8 *Hydrofluoric Acid* (sp gr 1.15)—Concentrated hydrofluoric acid (48 to 50 % HF).

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.

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7.9 *Ice Water*—Cool water to about 3 to 5°C by adding ice cubes, prepared from distilled water or water of equivalent purity.

7.10 *Iron (II) Ammonium Sulfate Solution* (approximately 0.1 N)—Dissolve 40 g of iron (II) ammonium sulfate, $(\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O})$ in dilute sulfuric acid (7.22). Transfer to a 1-L volumetric flask, dilute to volume with the same acid, and mix. Standardize against the standard potassium dichromate solution (7.13) using diphenylamine sulfonate as indicator (9.6).

7.11 *Perchloric Acid (1 + 1)*—Mix 1 volume of perchloric acid (70 %) (HClO_4) with 1 volume of water.

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

7.13 *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. Record the temperature at which this dilution was made.

7.14 *Potassium Permanganate Solution* (KMnO_4), 25 g/L.

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

7.16 *Sodium Carbonate Anhydrous Powder* (Na_2CO_3).

7.17 *Sodium Diphenylaminesulfonate Solution*—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.

7.18 *Sodium Hydroxide Solution* (NaOH), 20 g/L.

7.19 *Sodium Peroxide* (Na_2O_2), dry powder.

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

7.21 *Sulfuric Acid (1+1)*—Mix 1 volume of concentrated sulfuric acid (H_2SO_4) with 1 volume of water.

7.22 *Sulfuric Acid (1 + 19)*—Mix 1 volume of concentrated sulfuric acid (H_2SO_4) with 19 volumes of water.

7.23 *Sulfuric Phosphoric Acid Mixture*—Pour 150 mL of phosphoric acid (7.12) into about 400 mL of water. While stirring, add 150 mL of sulfuric acid (7.20). Cool in a water bath and dilute with water to 1 L.

7.24 *Tin (II) Chloride Solution* (100 g/L)—Dissolve 100 g of crystalline tin (II) chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 200 mL of hydrochloric acid (7.3) by heating the solution on a water bath. Cool the solution and dilute with water to 1 L. This solution should be stored in a brown glass bottle with the addition of a small quantity of granular or mossy tin metal.

7.25 *Titanium (III) Chloride Solution (2 %)*—Dissolve 1 g of titanium sponge in about 30 mL of hydrochloric acid (7.3) in a covered beaker by heating on a steam bath. Cool the solution and dilute with water to 50 mL. Prepare fresh as needed. (If preferred, dilute 1 volume of commercial titanium (III) chloride solution (about 15 % w/v) with 7 volumes of hydrochloric acid (7.4)).

8. Sample Preparation

8.1 The final sample shall be pulverized to pass a No. 100 (150- μm) sieve in accordance with Practice E 877.

NOTE 1—To facilitate decomposition, some ores, such as specular hematite, require grinding to pass a No. 200 (75- μm) sieve.

9. Hazards

9.1 For precautions to be observed in the use of certain reagents and equipment in this test method, refer to Practices E 50.

10. Procedure

NOTE 2—If the procedure is based on acid decomposition, use 10.1. If the procedure is based on an alkaline fusion followed by the filtration of the leached melt, (samples containing more than 0.1 % vanadium or molybdenum, or both), use 10.2. If the procedure is based on an alkaline fusion, followed by acidification of the leached melt (samples containing less than 0.1 % of molybdenum or vanadium, or both), use 10.3.

10.1 Acid Decomposition:

10.1.1 Weigh approximately 0.40 g of sample into a small weighing bottle previously dried at about 105°C (Note 3). 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 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 sample taken for analysis.

NOTE 3—For samples of iron content greater than 68 %, weigh approximately 0.38 g.

10.1.2 Carry a reagent blank through all steps of the procedure.

10.1.3 Add 30 mL of hydrochloric acid (7.3), cover the beaker with a watch glass, and heat the solution gently without boiling until no further attack is apparent. Wash the watch glass and dilute to 50 mL with warm water. Filter the insoluble residue on a close-texture paper. Wash the residue with warm hydrochloric acid (7.7), 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. Start to evaporate this solution.

10.1.4 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 sulfuric acid (7.21), add about 5 mL of hydrofluoric acid (7.8), and heat gently to remove silica and sulfuric acid. Add to the cold crucible 2 g of potassium pyrosulfate (7.15), heat gently at first, then strongly until a clear melt is obtained. Cool, place the crucible in a 250-mL beaker, add about 25 mL of water and about 5 mL of hydrochloric acid (7.3), and warm to dissolve the melt. Remove and wash the crucible.

10.1.5 Adjust the solution to slight alkalinity with ammonia solution (7.2). Heat to coagulate the precipitate, filter on a coarse-texture paper, and wash several times with hot water. Discard the filtrate.

10.1.6 Place the beaker containing the main solution under the funnel and dissolve the precipitate on the filter paper by pouring over it 10 mL of hot hydrochloric acid (7.5), wash the filter, first six to eight times with warm hydrochloric acid (7.7), then twice with hot water. Evaporate the combined filtrates at low heat to a volume of about 30 mL and continue with 10.4.

10.2 *Fusion Decomposition and Filtration of Leached Melt* (Note 2):



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