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Designation: E 878 – 01

Standard Test Method for Determination of Titanium in Iron Ores and Related Materials by Diantipyrylmethane Ultraviolet Spectrometry[1]

This standard is issued under the fixed designation E 878; 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 titanium in iron ores, concentrates, and agglomerates in the concentration range from 0.01 to 6.0 % titanium.

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 Water[2]

E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials[3]

E 135[3] Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E 877 Practice for Sampling and Sample Preparation of Iron Ores[4]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[4]

3. Terminology

3.1 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, nitric, and sulfuric acids, or by sintering with sodium peroxide, or by fusion with sodium tetraborate and sodium carbonate. Iron is reduced in an acid medium with ascorbic acid, the color is developed with diantipyrylmethane, and the absorbance is measured at approximately 385 nm.

5. Significance and Use

5.1 This test method is intended to be used for compliance with compositional specifications for titanium 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.

6. Interferences

6.1 None of the elements normally found in iron ores interfere.

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 *Ascorbic Acid Solution* (10 g/100 mL) ($C_6H_8O_6$)—Dissolve 10 g of ascorbic acid ($C_6H_8O_6$) in water and dilute to 100 mL. Prepare fresh as needed.

7.4 *Diantipyrylmethane Solution* (15 g/L) $C_{23}H_{24}O\ {}_2N_4 \cdot H_2O$—Dissolve 15 g of the reagent in about 300 mL of water

[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 11.01.

[3] *Annual Book of ASTM Standards*, Vol 03.05.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[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.

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and 30 mL of (H_2SO_4) (1 + 1) (7.10) and dilute to 1 L with water. If a residue remains, filter and store the filtrate in a brown bottle.

7.5 *Ferric Ammonium Sulfate* (100 g/L)—Dissolve 100 g of ferric ammonium sulfate ($Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4$ in 800 mL of water containing 5 mL of H_2SO_4 (1 + 1) (7.11) and dilute to 1 L with water.

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

7.7 *Hydrochloric Acid* (1 + 4)—Mix 1 volume HCl with 4 volumes of water.

7.8 *Potassium Pyrosulfate* ($K_2S_2O_7$).

7.9 *Sodium Tetraborate (Anhydrous)* ($Na_2B_4O_7$)—Dry the commercial sodium tetraborate at 60 to 70°C, then at 160°C, and finally calcine at 400°C.

7.10 *Sodium Tetraborate/Sodium Carbonate* ($Na_2B_4O_7$/Na_2CO_3) *Fusion Mixture*— Mix 1 part of $Na_2B_4O_7$ and 1 part of Na_2CO_3 and store in an airtight container.

7.11 *Sulfuric Acid* (1 + 1)—Carefully pour 1 volume of concentrated sulfuric acid (H_2SO_4) into 1 volume of water.

7.12 *Sulfuric Acid* (1 + 9)—Carefully pour 1 volume of H_2SO_4 into 9 volumes of water.

7.13 *Sulfuric Acid* (2 + 98)—Carefully pour 2 volumes of H_2SO_4 into 98 volumes of water.

7.14 *Standard Titanium Solution*:

7.14.1 *Solution A* (1 mL = 0.1 mg Ti)—Transfer 0.1670 g of TiO_2 (previously calcined at 900°C) to a platinum crucible, add 3 to 4 g of $K_2S_2O_7$, cover, and fuse at a temperature of 600°C until a clear melt is obtained. Place the cooled crucible and cover in a 250-mL beaker, add 50 to 60 mL of H_2SO_4 (1 + 9) (7.12), and heat to dissolve the melt. Wash crucible and cover with H_2SO_4 (1 + 9) (7.12) and remove, adding the washings to the 250-mL beaker. Transfer the solution of a 1-L volumetric flask, dilute to volume with H_2SO_4 (1 + 9) (7.12), and mix.

7.14.2 *Solution B* (1 mL = 0.02 mg Ti)—Transfer 50.0 mL of standard titanium Solution A to a 250-mL volumetric flask, dilute to volume with H_2SO_4 (1 + 9) (7.12), and mix.

8. Hazards

8.1 For precautions to be observed in this test method, refer to Practice E 50.

9. Sampling and Sample Preparation

9.1 *Sampling*—The gross sample shall be collected and prepared in accordance with Practice E 877.

9.2 *Sample Preparation*—Pulverize the laboratory sample to pass a No. 100 (150-m) sieve.

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

10. Procedure

NOTE 2—If the procedure is based on acid decomposition, use steps in 10.1. If the procedure is based on alkaline sintering, use steps in 10.2. If the procedure is based on alkaline fusion, use steps in 10.3.

10.1 *Acid Decomposition*:

10.1.1 Weigh approximately the amount of the test sample specified in the table below into a small weighing bottle previously dried at 150°C.

Ti content, %	Weight of test portion, g	Amount of sulfuric acid to be added in 9.1, mL	Aliquot, mL
0.01–0.1	1.0	20	20
0.1–0.3	1.0	20	10
0.3–1.0	0.5	10	5
1.0–6.0	0.1	10	5

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.

10.1.2 Carry a reagent blank through all steps of the procedure, starting with 10.1.3.

10.1.3 *Decomposition of Sample*—Moisten the test sample with a few milliliters of water, add 30 mL of hydrochloric acid, cover, and digest below the boiling point until no further attack is apparent. Add 5 mL of nitric acid and 10 to 20 mL of sulfuric acid (see amounts specified in 10.1.1) evaporate slowly to fumes of H_2SO_4, then heat strongly for 10 min. Allow the

TABLE 1 Grand Means and Precision of Titanium Content of the Test Samples as Determined by the Method Described Using Various Decomposition Methods

Sample No.	Decomposition Methods	Grand Mean *X*, %	Repeatability *r*, %	Permissible Tolerance *P*, %	Standard Deviation	
					Within-Laboratories σ_r, %	Between-Laboratories σ_L, %
76-3	Acid	3.7944	0.0788	0.1706	0.0285	0.0582
76-3	Sintering	3.8137	0.0848	0.2765	0.0306	0.0974
76-3	Fusion	3.8122	0.0785	0.1995	0.0283	0.0692
76-16	Acid	0.0399	0.0023	0.0042	0.0008	0.0014
76-16	Sintering	0.0402	0.0026	0.0051	0.0009	0.0017
76-16	Fusion	0.0402	0.0015	0.0034	0.0005	0.0012
76-17	Acid	0.1602	0.0032	0.0102	0.0012	0.0036
76-17	Sintering	0.1625	0.0049	0.0133	0.0018	0.0046
76-17	Fusion	0.1608	0.0055	0.0129	0.0020	0.0044
76-18	Acid	0.1796	0.0049	0.0081	0.0018	0.0027
76-18	Sintering	...	...	...	...	...
76-18	Fusion	0.1856	0.0090	0.0159	0.0032	0.0053

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