~~Designation:E878–01(Reapproved2005)~~ Designation: E878 – 11

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 E878; 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

~~1.1This 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~~

1.1 This test method covers the determination of titanium in iron ores, concentrates, and agglomerates in the compositional range from 0.01 % to 6.0 % titanium.

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 g/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 test method has been evaluated in accordance with Practice E1601 and Guide E1763. Unless otherwise noted in 13, the lower limit in the scope of each method specifies the lowest analyte content that may be analyzed with acceptable error (defined as a nominal 5 % risk of obtaining a 50 % or larger relative difference in results on the same test sample in two laboratories).

1.4 *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:*[2]

D1193 Specification for Reagent Water

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

E877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition

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

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods

3. Terminology

3.1 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, 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.

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[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.

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 E882.

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.[3] 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~~ 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 *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 and 30 mL of (H_2SO_4) ~~(1+1) (7.10)~~(1 + 1) 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~~$Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4$ 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~~*(1 + 1) and dilute to 1 L with water.*

7.6 *Potassium Pyrosulfate* ($K_2S_2O_7$).

~~7.9~~

7.7 *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~~*—Dry the commercial sodium tetraborate at 60 °C to 70 °C, then at 160 °C, and finally calcine at 400 °C.*

7.8 *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~~

7.9 *Standard Titanium Solution* :

~~7.14.1~~

7.9.1 *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 g 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 into a 250-mL beaker, add 50 mL to 60 mL of H_2SO_4 ~~(1+9) (7.12),~~(1 + 9), and heat to dissolve the melt. Wash crucible and cover with H_2SO_4 ~~(1+9) (7.12)~~(1 + 9) 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~~*(1 + 9), and mix.*

7.9.2 *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.~~ (1 + 9), and mix.

8. Hazards

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

9. Sampling and Sample Preparation

9.1 *Sampling*—The gross sample shall be collected and prepared in accordance with Practice E877.

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

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[3] *Reagent Chemicals, American Chemical Society Specifications*~~,~~ , American Chemical Society, Washington, ~~DC, www.chemistry.org.~~ DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary*~~,~~, U.S. ~~Pharmacopeial~~Pharmaceutical Convention, Inc. (USPC), Rockville, ~~MD, http://www.usp.org.~~MD.