Designation: E878 – 20

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

1.1 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 (mas/mass) of the form g/100 g.

1.2 *Units*—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 Section 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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
E877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition and Physical Properties
E882 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 (Withdrawn 2015)[3]

3. Terminology

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Relative Standard Deviation (RSD), n*—the RSD tells you whether the "regular" standard deviation is a small or large quantity when compared to the mean for the data set.

4. Summary of Test Method

4.1 The sample is decomposed by treatment with HCl, HNO_3, and H_2SO_4, or by sintering with sodium peroxide, or by

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

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 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 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.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall 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 *Ascorbic Acid Solution* (10 g/100 mL)—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) ($C_{23}H_{24}N_4O_2 \cdot H_2O$)—Dissolve 15 g of the reagent in about 300 mL of water and 30 mL of H_2SO_4 (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 $NH_4Fe(SO_4)_2 \cdot 12H_2O$ in 800 mL of water containing 5 mL of H_2SO_4 (1 + 1) and dilute to 1 L with water.

7.6 *Potassium Pyrosulfate* ($K_2S_2O_7$).

7.7 *Sodium Peroxide* (Na_2O_2).

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

7.9 *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.

7.10 *Titanium Calibration Solution:*

7.10.1 *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), and heat to dissolve the melt. Wash crucible and cover with H_2SO_4 (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), and mix.

7.10.2 *Solution B (1 mL = 0.02 mg Ti)*—Transfer 50.0 mL of titanium Solution A to a 250-mL volumetric flask, dilute to volume with H_2SO_4 (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 150-µm (No. 100) sieve.

NOTE 2—To facilitate decomposition, some ores such as specular hematite require grinding to pass a 75-µm (No. 200) sieve.

10. Procedure

10.1 If the procedure is based on acid decomposition, use steps in 10.2. If the procedure is based on alkaline sintering, use steps in 10.3. If the procedure is based on alkaline fusion, use steps in 10.4.

10.2 *Acid Decomposition:*

10.2.1 Weigh approximately the amount of the test sample specified in the Table 1 below into a small weighing bottle previously dried at 150 °C. Dry the bottle and contents for 1 h at 105 °C 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 loss of mass. 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 masses is the mass of the test sample taken for analysis.

10.2.2 Carry a reagent blank through all steps of the procedure, starting with 10.2.3.

TABLE 1 Amounts

Ti content, %	Mass of test sample, g	Amount of H_2SO_4 to be added in 10.2.3, 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

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

10.2.3 *Decomposition of Sample*—Moisten the test sample with a few milliliters of water, add 30 mL of HCl, cover, and digest below the boiling point until no further attack is apparent. Add 5 mL of HNO_3 and 10 mL to 20 mL of H_2SO_4 (see amounts specified in Table 1), evaporate slowly to fumes of H_2SO_4, then heat strongly for 10 min. Allow the solution to cool, add slowly 50 mL of water and 20 mL of HCl, and warm until soluble salts are in solution.

10.2.4 Filter on a fine-textured filter paper and collect the filtrate in a 250-mL beaker. Transfer the residue quantitatively to the filter paper and wash the filter paper two times or three times with hot H_2SO_4 (2 + 98) and two times or three times with hot water. Reserve the filtrate.

10.2.5 *Treatment of Insoluble Matter*—Ignite the paper and residue in a platinum crucible. Cool, moisten with several drops of water, add 3 drops or 4 drops of H_2SO_4 (1 + 1) and 10 mL of HF. Evaporate slowly to expel silica and excess of H_2SO_4. Cool, add to the residue about 2 g of potassium pyrosulfate, cover the crucible, and fuse over a burner (approximately 500 °C) until a clear melt is obtained.

10.2.6 Dissolve the cool melt in the reserved filtrate from 10.2.4, remove, and wash the crucible and cover, adding the washings to the 250-mL beaker. Transfer the solution to a 200-mL volumetric flask, dilute to volume, and mix. Continue as directed in 10.5.

10.3 *Alkaline Sintering Decomposition:*

10.3.1 Transfer a sample mass as directed in Table 1 into a small weighing bottle previously dried at 150 °C. Transfer the test sample to a 40-mL nickel crucible.

10.3.2 Carry a reagent blank through all steps of the procedure starting with 10.3.3.

10.3.3 Add 3 g of sodium peroxide and mix using a platinum or nickel spatula. Place the crucible for several minutes at the entrance of a muffle furnace set at 400 °C, then place the crucible inside the furnace for about 1 h, for sintering. Remove the crucible and allow to cool.

10.3.4 Transfer the sintered mass to a 250-mL beaker, cover, and add about 75 mL of water. Wash the crucible once with water and once with HCl (1 + 4), adding the washings to the beaker. Acidify carefully with 30 mL of HCl, cover the beaker, and heat gently until a clear solution is obtained. Add 20 mL of H_2SO_4 (1 + 1) and evaporate slowly to fumes of H_2SO_4; then heat strongly for 10 min. Allow the solution to cool, add slowly 50 mL of water and 20 mL of HCl, and warm until soluble salts are in solution.

10.3.5 Continue as described in 10.2.4 – 10.2.6. Finally conduct spectrophotometric measurements as described in 10.5.

10.4 *Alkaline Fusion Decomposition:*

10.4.1 Transfer a test sample mass as directed in Table 1 into a small weighing bottle previously dried at 150 °C. Transfer the sample to a platinum crucible.

10.4.2 Carry a reagent blank through all steps of the procedure starting with 10.4.3.

10.4.3 Add 5 g of fusion mixture (7.9) and mix, using a platinum or nickel spatula. Cover the crucible and heat in a muffle furnace, first gently at 600 °C and finally for 10 min at 1000 °C to 1050 °C. Remove the crucible and swirl cautiously to cause the cooling melt to solidify in a thin layer on the walls of the crucible.

10.4.4 Place the cooled crucible and cover into a 250-mL beaker and add 100 mL of HCl (1 + 4). Heat gently to dissolve the melt, remove, and wash the crucible, cover, and police adding the washing to the 250-mL beaker.

10.4.5 Transfer the solution to a 200-mL volumetric flask, dilute to volume, and mix. Continue as directed in 10.5.

10.5 *Preparation of Test Solution for Spectrophotometric Measurements*—Using a pipet, transfer an aliquot of the test solution and the blank solution as directed in Table 1 and transfer into 100-mL volumetric flasks. Add 5 mL of ferric ammonium sulfate solution (7.5), and 10 mL of ascorbic acid solution (7.3), and mix. Add 15 mL of HCl (1 + 1) and 30 mL of diantipyrylmethane solution (7.4), dilute to volume, and mix. Allow the solution to stand for at least 10 min.

10.6 *Preparation of Calibration Solutions for Spectrophotometric Measurements*—Using a pipet, transfer (0.0, 1.0, 3.0, 5.0, 7.0, and 10.0) mL of the titanium Solution B (7.10.2) to six 100-mL volumetric flasks, add 5 mL of ferric ammonium sulfate solution (7.5) and 10 mL of ascorbic acid solution (7.3), and mix. Add 15 mL of HCl (1 + 1) and 30 mL of diantipyrylmethane solution (7.4), dilute to volume, and mix. Allow the solution to stand for at least 10 min.

11. Spectrophotometry

11.1 Using water as the reference solution and a 1-cm cell (see Note 3), adjust the spectrophotometer to the initial setting of 100 % transmittance (0.0 absorbance), at a wavelength of 385 nm. Take spectrophotometric readings of the blank, calibration, and test solutions under the same measuring conditions.

11.2 *Preparation of Calibration Curve*—Subtract the average absorbance of the 0-mL titanium calibration solution from the average absorbance of each calibration solution and plot the net absorbance against milligrams of titanium per 100 mL of solution.

11.3 *Spectrophotometric Range*—The recommended concentration range is from 0.03 mg to 0.2 mg in 100 mL using a cell depth of 1 cm.

NOTE 3—Cells having other dimensions may be used, provided suitable adjustments can be made in the amount of sample and reagent used.

12. Calculation

12.1 Subtract the average absorbance of the reagent blank solution from the average absorbance of each of the test solutions. Convert the net absorbance of the test solutions to milligrams of titanium by means of the calibration curve. Calculate the titanium content as follows:

$$\text{Titanium, \%} = \frac{0.1A}{B} \qquad (1)$$

where:

A = titanium found in the aliquot used, mg, and
B = test sample mass in the aliquot, g.