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Standard Test Method for Determination of Phosphorus in Iron Ores by ~~Photometric Method~~Phospho-Molybdenum-Blue Spectrophotometry[1]

This standard is issued under the fixed designation E1070; 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 phosphorus in iron ores, concentrates, and agglomerates in the concentration range from 0.005 to 1.0 % phosphorus.

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~~

1.3 This test method has been evaluated in accordance with Practice E1601 and Guide E1763. Unless otherwise noted in the precision and bias section, 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

E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

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 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

4. Summary of Test Method

4.1 The sample is fused in a zirconium crucible with sodium peroxide. The melt is dissolved in water and hydrochloric acid. In a suitable aliquot, the molybdenum blue complex is formed by the addition of ammonium molybdate-hydrazine sulfate solution. The absorbance of the phospho-molybdenum-blue complex is measured at 725 nm.

5. Significance and Use

5.1 This test method for the analysis of iron ore concentrates and agglomerates is primarily intended as a referee method to test for compliance with compositional specifications. It is assumed that users of this test method will be trained analysts capable of

[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.

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 shall be followed, such as those described in Guide E882.

5.2 The determination of this element is needed for international trade and primary iron and steel making.

6. Interferences

6.1 Elements normally found in iron ores do not interfere excepting arsenic giving positive interference (0.01 % As = 0.001 % P).

7. Apparatus

7.1 *Zirconium Crucible*, 50 mL capacity.

7.2 *Spectrophotometer*—Visible spectrophotometer capable of measuring absorbance at the 725 nm wavelength using a 1-cm path length cell in accordance with Practice E60.

8. Reagents and Materials

8.1 *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.

8.2 *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.

8.3 *Ammonium Molybdate Solution (20 g/L)*—To 500 mL of water, add cautiously and slowly 300 mL of H_2SO_4 and cool. Add 20 g of ammonium molybdate $(NH_4)_6$-Mo_7O_{24}·$4H_2O$. Stir to dissolve and dilute to 1 L with water.

8.4 *Fusion Blank*—Dissolve 4 g of Na_2O_2 in 40 mL of water in a 250-mL beaker. Add 30 mL of HCl. Boil for 2 min. Cool and dilute to 100 mL with water in a volumetric flask. Prepare fresh as needed.

8.5 *Hydrazine Sulfate (1.5 g/L)*—Dissolve 0.15 g of hydrazine sulfate NH_2·NH_2·H_2SO_4 in water and dilute to 100 mL with water. Prepare fresh as needed.

8.6 *Molybdate* (5 g/L) *Hydrazine Sulfate* (0.15 g/L) *Solution*—Add 50 mL of ammonium molybdate solution (20 g/L) to 100 mL of water. Add 20 mL of hydrazine sulfate solution (1.5 g/L) and dilute to 200 mL with water.

NOTE 1—This solution should be prepared ~~freshly~~ within 30 min of use in a quantity appropriate for the number of tests being made.

8.7 *Sodium Peroxide (Na_2O_2)*—Use caution when using peroxide.

8.8 *Sodium Sulfite Solution (100 g/L)*—Dissolve 10 g of sodium sulfite (Na_2SO_3) in water and dilute to 100 mL with water.

8.9 *Standard Phosphorus Solution*—Dry anhydrous disodium phosphate (Na_2HPO_4) at 105 °C for 2 h and after desiccation, dissolve 0.2292 g of the reagent in 200 mL of water. Dilute to 1 L with water in a volumetric flask and mix. This solution, A, provides 1 mL = 50 µg P. Transfer 10.00 mL of solution A into a 50-mL volumetric flask, dilute to mark with water, and mix. This solution, B, provides the standard phosphorus solution for calibration 1 mL = 10 µg P.

9. Hazards

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

10. Sampling and Sample Preparation

10.1 Collect and prepare gross samples in accordance with Practice E877.

10.2 Pulverize the laboratory sample to pass a No. 100 (150-µm) sieve.

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

11. Calibration and Standardization

11.1 The recommended ~~concentration~~ range of phosphorus content is from 0.005 mg to 0.10 mg phosphorus in 50 mL of color solution using cell depth of 1 cm.

11.2 Into a series of five 150-mL beakers, transfer 10.0 mL of fusion blank solution (8.4) and then transfer (0, 0.50, 2.50) mL of standard B; 1.00 mL and 2.00 mL of standard A phosphorus solution (8.9) corresponding to (0, 5, 25, 50, and 100) µg of phosphorus respectively. To each beaker add 15 mL of sodium sulfite solution (8.8). Mix. Bring to a boil. Add 20 mL of

[3] ~~*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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, http://www.usp.org.~~ 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.