Designation: E1070 – 17

Standard Test Method for
Determination of Phosphorus in Iron Ores by 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 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 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.*

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

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 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 *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 (Na_2O_2). The melt is dissolved in water and HCl. 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 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

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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.

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.[4] 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 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 $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$. 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 $NH_2\cdot NH_2\cdot H_2SO_4$ 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.

8.6.1 This solution should be prepared 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 1—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 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 phosphorus solution (8.9); 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 molybdate-hydrazine sulfate solution (8.6). Bring to a boil. Simmer for 10 min in boiling water bath. Cool. Transfer into a 50-mL volumetric flask and dilute to the mark with water. Mix. This solution is stable for at least 2 h.

11.3 *Spectrophotometry*—Adjust the spectrophotometer to the initial setting, using water as the reference solution. While maintaining this setting, measure absorbance of the calibration standard and sample solutions at 725 nm in a 1-cm cell.

11.4 *Preparation of Calibration Curve*—Subtract the absorbance of the 0-mL phosphorus solution from the absorbance of each calibration solution and plot the net absorbance against µg of phosphorus in the color solution.

12. Procedure

12.1 Weigh approximately the amount of the prepared sample specified in the following table into a small weighing bottle previously dried at 150 °C:

P Content, %	Mass of Sample, g	Sample Aliquot, mL	Fusion Blank, mL
0.005 to 0.15	0.300	10.0	None
0.15 to 0.50	0.100	10.0	None
0.50 to 1.0	0.100	5.0	5.0

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[4] *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 the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Statistical Information

Sample	Average Phosphorus Content, %	Standard Deviation	Relative Standard Deviation, %	Number of Participating Laboratories	Number of Replicates
Japan 830-1 (0.125)[A]	0.1270	0.0035	2.75	8	24
NBS 690 (0.011)[A]	0.0085	0.0010	11.76	8	24
NBS 692 (0.039)[A]	0.0393	0.0022	5.61	8	24
NBS 693 (0.056)[A]	0.0554	0.0020	3.61	8	24

[A] *Certificate Value*—Phosphorus content, %.

Dry the 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 test sample to the nearest 0.1 mg. Repeat the drying and weighing until there is no further loss of mass. Transfer the test sample into a zirconium crucible and reweigh the capped bottle to the nearest 0.1 mg. The difference between the two masses is the mass of test sample taken for analysis. Add 2 g of Na_2O_2 to the crucible and mix with a dry, stainless steel spatula.

NOTE 2—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/100 g.

12.2 Fuse over a burner, swirling the crucible until the melt is cherry red and clear. Remove from the heat and swirl until the melt solidifies on the wall of the crucible.

12.3 Place the crucible in a 250-mL dry beaker and cool. Cover with a watch glass and add about 10 mL of water to the crucible. After the reaction ceases, transfer the crucible contents into the beaker, and wash the crucible with water. Add 15 mL of HCl to the crucible and transfer to the beaker. Rinse the crucible with water and add the rinsings to the beaker. Boil for 2 min. Cool. Transfer the contents and rinsings into a 50-mL volumetric flask. Dilute to volume with water and mix.

12.4 Transfer appropriate aliquot of the test solution and fusion blank aliquot (in accordance with 11.2) into a 150-mL beaker. Add 15 mL of sodium sulfite solution (100 g/L). Mix. Bring to a boil. Add 20 mL of molybdate (5 g/L) hydrazine sulfate (0.15 g/L) solution. Bring to a boil. Simmer for 10 min in a boiling water bath. Cool. Transfer into a 50-mL volumetric flask and dilute to the mark with water. Mix.

12.5 Measure absorbance at 725 nm in a 1-cm cell in the same manner as the calibration solutions.

13. Calculation

13.1 Subtract the absorbance of the 0-mL phosphorus solution from the absorbance of the test sample solutions. Determine µg phosphorus referring to the calibration curve.

13.2 Calculate the phosphorus content as follows:

$$\text{Phosphorus, \%} = \left(\frac{0.005A}{B \times C}\right) \quad (1)$$

where:

A = µg of phosphorus determined,
B = sample aliquot in milliliters (12.1), and
C = sample mass in grams (12.1).

13.3 Rounding of test results obtained using this test method shall be performed in accordance with Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification.

NOTE 3—As used in this test method, "percentage" or "%" refers to a mass fraction of the form (wt / wt %) (g / 100 g).

14. Precision and Bias[5]

14.1 *Precision*—Table 1 indicates the precision of the test method between laboratories and comparison with the certified reference materials.

14.2 *Bias*—The results obtained by the 8 laboratories on the certified reference iron ores, of varying composition, agree closely within narrow limits with the assigned certified phosphorus values, thus demonstrating absence of any measurable bias.

15. Keywords

15.1 agglomerates and related materials; concentrates; iron ores; phosphorus content

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[5] Supporting data are available from ASTM International Headquarters. Request RR:E16-1009.

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