



Standard Test Method for Determination of Phosphorus in Iron Ores by Photometric Method¹

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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 *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 Water²

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals³

E 60 Practice for Analysis of Metals, Ores, and Related Materials by Molecular Absorption Spectrometry³

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials³

E 877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials⁴

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory⁴

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E 135.

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.

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. 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 procedures will be followed. Appropriate quality control practices shall be followed, such as those described in Guide E 882.

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 Practices E 60.

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.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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

8.3 *Ammonium Molybdate Solution* (20 g/L)—To 500 mL of water, add cautiously and slowly 300 mL of H₂SO₄ and cool.

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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

Add 20 g of ammonium molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$. 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.

10. Sampling and Sample Preparation

10.1 Collect and prepare gross samples in accordance with Practice E 877.

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

TABLE 1 Statistical Information

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

^A Standard deviation.

^B Relative standard deviations in this test method are calculated respectively as follows:

$$\sigma = \sqrt{\frac{d^2}{(n-1)}}$$

$$v = \left(\frac{100}{\bar{x}}\right) \sqrt{\frac{d^2}{(n-1)}}$$

where:

\bar{x} = average concentration, %,

d = difference of the determination from the mean, and

n = number of determinations.

^C Certificate value—Phosphorus concentration, %.

8.5 *Hydrazine Sulfate* (1.5 g/L)—Dissolve 0.15 g of hydrazine sulfate $\text{NH}_2\text{NH}_2\cdot\text{H}_2\text{SO}_4$ 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 Practice E 50.

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 is from 0.005 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 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 *Photometry*—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 approximately 725 nm in a 1-cm cell.

11.4 *Preparation of Calibration Curve*—Subtract the absorbance of the 0-mL phosphorus standard solution from the absorbance of each standard 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, %	Weight 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

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

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) in 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 solution (0.15 g/L). 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 approximately 725 nm in a 1-cm cell in the same manner as the calibration standards.

13. Calculation

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

13.2 Calculate the percentage of phosphorus as follows:

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

where:

A = μg of phosphorus determined,

B = sample aliquot in millilitres (12.1), and

C = sample weight in grams (12.1).

14. Precision and Bias ⁶

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

⁶ Supporting data are available from ASTM Headquarters. Request RR: E16-1009.

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