



Standard Test Method for Phosphorus in Iron Ores (Titrimetric)¹

This standard is issued under the fixed designation E 278; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

^{ε1} NOTE—Editorial changes were made throughout in June 1997.

1. Scope

1.1 This test method covers the determination of phosphorus in iron ores, concentrates, and agglomerates.

1.2 This test method covers the determination of phosphorus in the concentration range from 0.01 to 1.00 %.

1.3 *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 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

E 877 Practice for Sampling and Sample Preparation of Iron Ores⁴

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

3. Summary of Test Method

3.1 The sample is dissolved in hydrochloric and nitric acids. After the addition of perchloric acid, the solution is evaporated to strong fumes to dehydrate the silica. The insoluble residue is filtered off, ignited, and treated for the recovery of any contained phosphorus. Ammonium molybdate is added to precipitate phosphomolybdate. The precipitate is filtered off and washed free from acid. It is then dissolved in an excess of standard sodium hydroxide solution. The excess sodium hydroxide is titrated with a standard solution of nitric acid using phenolphthalein as an indicator.

4. Significance and Use

4.1 This test method is intended to be used for compliance

with compositional specifications for phosphorus 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 shall be followed, such as those described in Guide E 882.

5. Interferences

5.1 Vanadium and arsenic, elements commonly found in iron ores, coprecipitate with the phosphorus. Provisions for their removal or elimination of their interference are included in this test method.

5.2 Titanium tends to form an insoluble compound with phosphorus and thus may cause low values for phosphorus. Provision for its removal is included in this test method.

6. Reagents

6.1 *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.

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

6.3 *Ammonium Molybdate Solution (Acidic)*—Reagent No. 102.

6.4 *Ammonium Nitrate* (NH₄NO₃).

6.5 *Ferric Chloride Solution*—Dissolve 0.3 g of pure iron wire in 25 mL of HCl (1 + 1). Oxidize by adding HNO₃ dropwise to the hot solution. Cool, add 25 mL of HCl, dilute to 1 L and mix.

6.6 *Ferrous Sulfate Solution*—Dissolve 100 g of ferrous sulfate (FeSO₄·7H₂O) in 1 L of sulfuric acid (H₂SO₄, 5 + 95).

¹ This test method is under the jurisdiction of ASTM Committee E-1 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 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.06.

⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."