



Standard Test Method for Determination of Phosphorus in Nickel, Ferronickel, and Nickel Alloys by the Phosphovanadomolybdate Molecular Absorption Spectrometric Method¹

This standard is issued under the fixed designation E 1917; 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.

1. Scope

1.1 This test method covers the determination of phosphorus in nickel, ferronickel, and nickel alloys in the 0.0007 % through 0.05 % range.

1.2 Arsenic, chromium, hafnium, niobium, silicon, tantalum, titanium, and tungsten interfere, but the interference can be avoided by complexation or volatilization (for Cr). The lowest phosphorus content (0.0007 %) can be reached only in samples with low contents of interfering elements.

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.* For specific hazards associated with the use of this practice see Practices E 50. Refer to specific warning notes given throughout this test method.

2. Referenced Documents

2.1 ASTM Standards:

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory³

E 1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analysis³

E 1601 Practice for Conducting Interlaboratory Study to Evaluate the Performance of an Analytical Method³

2.2 ISO Standards:

ISO 5725:1986 Precision of Test Methods—Determination of Repeatability and Reproducibility for a Standard Test

Method by Inter-laboratory Tests⁴

ISO 11400:1992(E) Nickel, Ferronickel and Nickel Alloys—Determination of Phosphorus Content—Phosphovanadomolybdate Molecular Absorption Spectrometric Method⁴

3. Summary of Test Method

3.1 The sample is dissolved in a mixture of hydrochloric acid and nitric acid. The solution is evaporated to perchloric acid fumes and chromium is removed as volatile chromyl chloride. Silicon and refractory elements are complexed with fluoride ions. The phosphorus is converted to phosphovanadomolybdic acid in a perchloric and nitric acid solution. The phosphovanadomolybdic acid is extracted with 2-methyl-2-pentanone in the presence of citric acid to complex arsenic. Absorbance is measured at 355 nm.

4. Significance and Use

4.1 This test method is used for the analysis of nickel, ferronickel, and nickel base alloy samples by molecular absorption spectrometry to check compliance with compositional specifications. It is assumed that all who use the procedure will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that the 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 E 882.

5. Apparatus

5.1 *Spectrophotometer*—Capable of measuring absorbance at a wavelength of 355 nm.

5.2 *Cells*—To fit spectrophotometer, having an optical path of 1 cm.

NOTE 1—Cells having other dimensions can be used, provided suitable adjustments can be made in the amount of sample and reagents used.

5.3 Plastic separatory funnels, 250 mL capacity.

¹ This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry of Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.08 on Nickel, Cobalt and High Temperature Alloys.

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² *Annual Book of ASTM Standards*, Vol 03.05.

³ *Annual Book of ASTM Standards*, Vol 03.06.

⁴ Available from American National Standards Institute, 11 West 42nd Street, New York, NY 10036.