Designation: ~~E1917 – 08~~ E1917 – 13

Standard Test Method for Determination of Phosphorus in Nickel, Ferronickel, and Nickel Alloys by Phosphovanadomolybdate ~~Molecular Absorption Spectrometry~~Spectrophotometry[1]

This standard is issued under the fixed designation E1917; 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 nickel, ferronickel, and nickel alloys in the range 0.0007 % to 0.05 %.

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

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.* For specific hazards associated with the use of this test method see Practices E50. Refer to specific warning notes given throughout this test method.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

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

2.2 *ISO Standards:*[3]

ISO 5725:1986 Precision of Test Methods—Determination of Repeatability and Reproducibility for a Standard Test Method by Inter-laboratory Tests

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

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 dissolved in a mixture of HCl and HNO_3. $HClO_4$ is added and the solution is evaporated to fumes of $HClO_4$ to remove chromium as volatile chromyl chloride. Silicon and refractory elements are complexed with fluoride ions through the addition of HF. Phosphorus is converted to phosphovanadomolybdic acid in an $HClO_4$ and HNO_3 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.

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

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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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

5. Significance and Use

5.1 This test method is used for the determination of phosphorus in nickel, ferronickel, and nickel 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 E882.

6. Apparatus

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

6.2 *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.

6.3 Plastic separatory funnels, 250-mL capacity.

7. Reagents

7.1 *Purity and Concentration of Reagents*—The purity and concentration of common chemical reagents and water shall conform to Practices E50. The reagents should be free of or contain only minimal amounts (< 0.1 µg/g) of phosphorus.

7.1.1 Verify the absence of phosphorus in the reagents using the blank test. Reagents giving high blank values are unsuitable and should not be used. The blank value for all reagents should be below 0.0005 % *P* calculated for a 1-g sample.

7.2 *Ammonium Metavanadate Solution*—Dissolve 2.5 g of ammonium metavanadate (NH_4VO_3) in water, dilute to 1 L, and mix.

7.3 *Citric Acid Solution*—Dissolve 500 g citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) in water, dilute to 1 L, and mix. Warm the solution if necessary to facilitate dissolution.

7.4 *Fluoroboric Acid Solution*—Disperse 75 g of boric acid (H_3BO_3) in 600 mL of hot water in a plastic beaker. Add 50 mL of HF (40 %) and dilute to 1 L. Digest over medium heat until the boric acid is dissolved. Store in a plastic bottle. The solution should be heated gently if the boric acid forms crystals. (**Warning—**HF and fluoroboric acid are extremely irritating and corrosive to skin and mucous membranes, producing severe skin burns that are slow to heal. In case of contact with skin, wash well with water and seek medical advice. When using HF and fluoroboric acid, always wear appropriate safety gear, such as goggles and gloves.)

7.5 *Hexaammonium Heptamolybdate Solution*—Dissolve 15 g of hexaammonium heptamolybdate tetrahydrate [$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$] in warm water and dilute to 100 mL. Prepare fresh solution each day. If high and unstable blank values appear, there might be a problem with the salt used. In such a case, switch to another lot.

7.6 *4-Methyl-2-pentanone*—Methylisobutyl ketone.

7.7 *Phosphorus Stock Calibration Solution (1.000 g/L)*—Transfer 4.3942 g of potassium dihydrogenorthophosphate (KH_2PO_4) (which has been previously dried at 110 °C to constant weight and cooled in a desiccator) to a 1-L volumetric flask. Dissolve in water, dilute to the mark, and mix.

7.8 *Phosphorus Calibration Solution (10 mg/L)*—Transfer 10.0 mL of the phosphorus stock calibration solution to a 1-L volumetric flask. Dilute to the mark and mix.

7.9 *Sodium Nitrite Solution (50 g/L)*—Dissolve 50 g of sodium nitrite ($NaNO_2$) in water and dilute to 1 L.

8. Sampling and Sample Preparation

8.1 The sampling shall be performed by normal procedures agreed upon between the parties, or in the event of a dispute, in accordance with the relevant standard, if one is available.

8.2 The laboratory sample is normally in the form of millings or drillings and no further preparation of the sample is necessary.

8.3 If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling operation, it shall be cleaned by washing it with high purity acetone, or other appropriate solvent, and dried in air.

8.4 If the sample contains particles or pieces of widely varying sizes, the test sample should be obtained by riffling.

TABLE 1 Weight of Test Portion of the Sample

Expected Phosphorus Content, %	Weight of Test Portion, g	Maximum concentration of the interfering elements, %					
		As	Hf	Nb	Ta	Ti	W
0.0005 to 0.010	1.0	0.05	0.1	1	0.1	2	2
0.002 to 0.04	0.25	0.2	0.5	5	0.5	10	8
0.005 to 0.050	0.10	0.5	1.5	10	1	25	25