**~~Designation:E1917–02~~ Designation: E 1917 – 08**

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

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

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

~~1.2Arsenic, 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~~

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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 ~~practice~~test method see Practices E 50. Refer to specific warning notes given throughout this test method.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

E 50 ~~Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals~~ Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

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

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory ~~E1452Practice for Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analysis~~

E 1601 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. ~~Summary of Test Method~~

~~3.1The sample is dissolved in a mixture of hydrochloric acid and nitric acid. The solution is evaporated to perchloric acid fumes~~

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

Current edition approved ~~Sept 10, 2002.~~Dec. 15, 2008. Published ~~June 2003.~~January 2009. Originally approved in 1997. Last previous edition approved in ~~1997~~2002 as ~~E 1917-97.~~E 1917 – 02.

[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, 11 West 42nd Street, New York, NY 10036.~~

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

~~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.~~

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 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.

## 5. Significance and Use

~~4.1This~~5.1 This test method is used for the ~~analysis~~determination of phosphorus in 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.~~

## 6. Apparatus

~~5.1~~

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

~~5.2~~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.

~~5.3Plastic separatory funnels, 250 mL capacity.~~

~~6.~~

6.3 Plastic separatory funnels, 250-mL capacity.

## 7. Reagents

~~6.1~~

7.1 *Purity and Concentration of Reagents*—The purity and concentration of common chemical reagents and water shall conform to Practices E 50. The reagents should be free of or contain only minimal amounts (< 0.1 µg/g) of phosphorus. ~~Calibration solutions shall be prepared in accordance with Practice E1452.~~

~~6~~7.1.1 Verify the absence of phosphorus in the reagents using the blank ~~test (9.6.1).~~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.~~

~~6.2~~*calculated for a 1-g sample.*

7.2 *Ammonium Metavanadate Solution*—Dissolve 2.5 g of ammonium metavanadate ($NH_4VO_3$) ~~in water and dilute to 1 L.~~ ~~6.3~~*) 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 and~~ water, dilute to 1 L, and mix. Warm the solution if necessary to facilitate dissolution.

~~6.4~~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 ~~gently~~ heated gently if the boric acid forms crystals. ~~Note2—(Warning:~~**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.)

**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 |

**TABLE 2 Nominal Composition of Nickel and Ferro-nickel Samples, %**

| Test Material | P | As | Cr | Fe | Si | Ni |
|---|---|---|---|---|---|---|
| Ni #1 | 0.00005 | - | - | < 0.01 | - | balance |
| Ni #2 | 0.0008 | - | - | balance | - | balance |
| Fe-Ni #1 | 0.01 | < 0.001 | 0.5 | balance | 0.5 | 25 |
| Fe-Ni #2 | 0.01 | 0.1 | 4.5 | balance | 5 | 25 |
| Fe-Ni #3 | 0.045 | 0.001 | 0.5 | balance | 0.6 | 25 |
| Fe-Ni #4 | 0.045 | 0.1 | 4.5 | balance | 5 | 25 |

**TABLE 3 Nominal Composition of Nickel Alloy Samples, %**

| Sample | P | Co | Cr | Cu | Fe | Mo | Ni | Nb | W |
|---|---|---|---|---|---|---|---|---|---|
| 4D-7 | 0.01 | - | - | 32 | 1 | - | 65 | - | - |
| 4D-8 | 0.01 | - | 21 | - | 4 | 9 | 62 | 3 | - |
| 4D-9 | 0.02 | - | 19 | - | 19 | 3 | 53 | 5 | - |
| 4D-10 | 0.02 | - | 20 | - | 46 | - | 31 | - | - |
| 4D-11 | 0.01 | - | 21 | - | 20 | 8 | 47 | - | - |
| 4D-12[A] | 0.005 | 42 | 21 | - | 2 | 4 | 20 | 4 | 4 |

[A] Standard Reference alloy BAM 328-1, certified at 0.005 % *P* was used as sample 4D-12.

**TABLE 4 Results of Statistical Analysis**

| Test Material[A] | Mean, % | Repeatability ~~SD~~ Index ($S_{Pr}$, ~~ISO 5725~~actice E 1601)[B] | Reproducibility ~~SD ($S_R$,~~ ~~ISO~~ 5725ndex (Practice E 1601)~~C~~ | ~~Reproducibility (R, ISO 5725)~~[D] | $R_{rel\ \%}$ |
|---|---|---|---|---|---|
| ~~Ni #2~~ | ~~0.00091~~ | ~~0.00007~~ | ~~0.00012~~ | ~~0.0004~~ | ~~44~~ |
| Ni #2 | 0.00091 | 0.0002 | 0.00012 | ~~0.0004~~ | ~~44~~ |
| ~~4D-12[E]~~ | ~~0.0054~~ | ~~0.0001~~ | ~~0.0008~~ | ~~0.002~~240 | |
| 4D-12[B] | 0.0054 | 0.0004 | 0.0008 | ~~0.0~~022 | |
| ~~4D-8~~ | ~~0.0089~~ | ~~0.0009~~ | ~~0.00150.0050~~ | ~~56~~ | |
| 4D-8 | 0.0089 | 0.0024 | 0.0050~~.0050~~ | ~~56~~ | |
| ~~FeNi #1~~ | ~~0.0100~~ | ~~0.0004~~ | ~~0.0002~~ | ~~0.0014~~ | 14 |
| FeNi #1 | 0.0100 | 0.0012 | 0.0092 | ~~0.0014~~ | ~~14~~ |
| ~~FeNi #2~~ | ~~0.0100~~ | ~~0.0004~~ | - | 0.0012 | 12 |
| FeNi #2 | 0.0100 | ~~0.0004~~ | - | 0.0012 | 0.0012 |
| ~~4D-7~~ | ~~0.0120~~ | ~~0.0007~~ | ~~0.0007~~ | ~~0.002~~621 | |
| 4D-7 | 0.0120 | 0.0016 | 0.0097 | ~~0.00~~26 | |
| ~~4D-11~~ | ~~0.0135~~ | ~~0.0006~~ | ~~0.0008~~ | ~~0.002~~720 | |
| 4D-11 | 0.0135 | 0.0016 | 0.0098 | ~~0.00~~27 | |
| ~~4D-9~~ | ~~0.0148~~ | ~~0.0008~~ | ~~0.0006~~ | ~~0.00~~28 | ~~18~~ |
| 4D-9 | 0.0148 | 0.0023 | 0.0096 | ~~0.00~~28 | ~~18~~ |
| ~~4D-10~~ | ~~0.0185~~ | ~~0.0003~~ | ~~0.0007~~ | ~~0.002~~211 | |
| 4D-10 | 0.0185 | 0.0010 | 0.0097 | ~~0.00~~22 | |
| ~~FeNi #4~~ | ~~0.0425~~ | ~~0.0010~~ | ~~0.0010~~ | ~~0.00~~388.9 | |
| FeNi #4 | 0.0425 | 0.0027 | 0.0010 | ~~0.00~~38 | |
| ~~FeNi #3~~ | ~~0.0437~~ | ~~0.0014~~ | 0.0021 | 0.006815 | |
| FeNi #3 | 0.0437 | ~~0.0014~~ | 0.0021 | 0.0068 | |

[A] Material ~~C~~ compositions are summarized in Table 2 and Table 3.
[B] ~~Equivalent to minimum standard deviation, $s_M$, (Practice E1601).~~
[C] Equivalent to reproducibility standard deviation, $s_R$, (Practice E1601)~~.~~
[D] ~~Equivalent to reproducibility index, R, (Practice E1601).~~
[E] Standard Reference alloy BAM 328-1, certified at 0.005 % *P* was used as sample 4D-12.



6.57.5 *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.

6.67.6 *4-mMethyl-2-pentanone*—mMethylisobutyl ketone.

6.77.7 *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.

6.87.8 *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 ~~with water~~ and mix.

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