



Designation: **E1587 – 10** E1587 – 17

## Standard Test Methods for Chemical Analysis of Refined Nickel<sup>1</sup>

This standard is issued under the fixed designation E1587; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 These test methods apply to the chemical analysis of refined nickel and other forms of metallic nickel having chemical compositions within the following limits:

Element	Weight, % Mass Fraction, %
Antimony, less than	0.005
Arsenic, less than	0.005
Bismuth, less than	0.01
Cadmium, less than	0.0025
Carbon, max	0.03
Cobalt, max	1.00
Copper, max	1.00
Hydrogen, max	0.003
Iron, max	0.15
Lead, less than	0.01
Manganese, less than	0.20
Nickel, min	98.0
Nitrogen, less than	0.50
Oxygen, less than	0.03
Phosphorus, less than	0.005
Selenium, less than	0.0010
Silicon, less than	0.005
Silver, less than	0.01
Sulfur, max	0.01
Tellurium, less than	0.0010
Thallium, less than	0.0010
Tin, less than	0.005
Zinc, less than	0.015

1.2 These test methods may be used to determine the following elements by the methods indicated below:

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.08 on Ni and Co and High Temperature Alloys.

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Test Methods	Sections
Antimony, Arsenic, Bismuth, Cadmium, Lead, Selenium, Silver, Tellurium, Tin, and Thallium by the Graphite Furnace Atomic Absorption Spectrometric Method	21 – 31
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Test Methods	Sections
Nitrogen by the Inert Gas Fusion Thermal Conductivity Method (Refer to Test Methods <del>E1019</del> )	45
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Sulfur by the Methylene Blue Spectrophotometric Method After Generation of Hydrogen Sulfide	34–44
Sulfur by the Methylene Blue Spectrophotometric Method After Generation of Hydrogen Sulfide	<u>32 – 42</u>

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 precautions, see Section 6.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

[D1193 Specification for Reagent Water](#)

[E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications](#)

[E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials](#)

[E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry](#)

[E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials](#)

[E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques](#)

[E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry \(Withdrawn 2004\)<sup>3</sup>](#)

[E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method](#)

[E1917 Test Method for Determination of Phosphorus in Nickel, Ferronickel, and Nickel Alloys by Phosphovanadomolybdate Spectrophotometry](#)

### 2.2 ISO Standard:<sup>4</sup>

[ISO 5725 Precision of Test Methods—Determination of Repeatability and Reproducibility by Interlaboratory Tests](#)

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

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

### 3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology [E135](#).

### 4. Significance and Use

4.1 These test methods are primarily intended to test refined nickel metal for compliance with compositional specifications. It is assumed that all who use these test methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that the analytical work will be performed in a properly equipped laboratory under appropriate quality control practices.

### 5. Apparatus, Reagents, and Instrumental Practices

#### 5.1 Apparatus:

5.1.1 Special apparatus and reagents required for each determination are listed in the Apparatus section of each test method.

5.1.2 Glass storage containers shall be of borosilicate glass.

5.1.3 Plastic containers shall be polyethylene or preferably polytetrafluoroethylene (PTFE).

#### 5.2 Reagents:

5.2.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents used in these test methods shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>5</sup> Other chemical grades may be used, provided it is first ascertained that they are the reagent is of sufficiently high purity to permit their use without adversely affecting the expected performance of the determination, as indicated in each Precision and Bias section lessening the accuracy of the determination.

5.2.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification [D1193](#).

5.2.3 Reagents and their preparation are described in the Reagents section in each test method.

5.2.4 Instructions for the preparation of standard solutions used in these test methods frequently call for measuring exact weights masses of substances of known composition so that the concentrations of the resulting standard stock solutions can be expressed using simple numbers. Small variations from these specified quantities are acceptable, provided that the true weighed masses are used to calculate the concentration of the prepared solutions and then these calculated values are used throughout the test methods.

5.3 *Instrumental Practices*—Information on the use of some instrumental techniques employed in these test methods are described in Practice [E60](#) and in Guide [E1024](#).

### 6. Hazards

6.1 For precautions to be observed in the use of certain reagents and equipment in these test methods, refer to Practices [E50](#).

6.2 Where appropriate, specific precautionary information is given in the Hazards sections and in special warning paragraphs.

### 7. Sampling

7.1 Sampling shall be carried out by a mutually acceptable method.

7.2 The laboratory sample normally is in the form of a powder, granules, millings, or drillings and no further preparation is necessary.

7.3 If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it may be cleaned by washing with high-purity acetone and drying in air.

7.4 If the laboratory sample contains particles or pieces of widely varying sizes, the test sample should be obtained by riffing or coning and quartering techniques.

### 8. Rounding Calculated Values

8.1 Calculated values shall be rounded to the desired number of places in accordance with the rounding method in Practice [E29](#).

<sup>5</sup> Available from Fisher Scientific Co., 2000 Park Lane Dr., Pittsburgh, PA 15275, [www.fishersci.com](http://www.fishersci.com); 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 the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

# SILVER, BISMUTH, CADMIUM, COBALT, COPPER, IRON, MANGANESE, LEAD, AND ZINC BY THE-FLAME ATOMIC ABSORPTION SPECTROMETRIC METHODS SPECTROMETRY

## 9. Scope

9.1 This test method applies to the determination of the silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead, and zinc contents of refined, wrought, and cast nickel metal within the following ranges.

Element	Method A	Concentration Range, %	Method B
		Mass Fraction Range, %	
Silver	0.0002 to 0.01		...
Bismuth	0.0010 to 0.01		...
Cadmium	0.0002 to 0.0025		...
Cobalt	0.0010 to 0.01		0.01 to 1.00
Copper	0.0005 to 0.01		0.01 to 1.00
Iron	0.0025 to 0.01		0.01 to 0.15
Manganese	0.0005 to 0.01		0.01 to 0.20
Lead	0.0006 to 0.01		...
Zinc	0.0005 to 0.0025		0.001 to 0.015

9.2 This test method is applicable to the independent determination of any one or more of the elements listed without including all the elements specified in the standard calibration solutions.

9.3 The lower level for iron can be extended to less than 0.0025 % provided nickel metal containing less than 0.0001 % iron is used for preparation of standards calibration solutions.

9.4 The upper limit for the determination of cobalt and copper can be raised to 2 % by a minor modification to the test method. See For Note 6. test samples containing greater than 0.25 % and less than 2 % of cobalt or copper, further dilutions of the test solution with HNO<sub>3</sub> (1 + 19) may be made. The nickel content of the calibration solutions should be matched with those of the test solutions.

9.5 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

## 10. Summary of Test Method

10.1 The sample is dissolved in dilute HNO<sub>3</sub>, excess acid is evaporated, and the solution diluted to a known volume. The test solution is aspirated into the air/acetylene flame of an atomic absorption spectrometer. The absorption of the resonance line energy from the spectrum of each element is measured and compared with that from a set of calibration solutions of the same element in a matched nickel matrix.

## 11. Interferences

11.1 Elements ordinarily present in nickel metal do not present spectral interferences in the atomic absorption analysis.

11.2 For the determination of silver, take care to avoid contamination of the sample and calibration solutions with chloride.

11.3 Potential background absorption interference is eliminated by use of matched matrix standards calibration solutions prepared from high-purity nickel metal. See Note 7.

NOTE 1—In this test method, any effect of nonspecific absorption and light scatter is compensated for by matching the matrix of the calibration solutions with the test solutions. Also, since the same lot of HNO<sub>3</sub> is used for both calibration and test solutions, the reagent blank is incorporated in the calibration curve. Thus, the calibration curve may not pass through the origin.

## 12. Apparatus

12.1 *Atomic Absorption Spectrometer:*

12.1.1 The atomic absorption spectrometer used in this test method should meet the instrument performance parameters in accordance with Guide E1024.

12.1.2 The instrument shall be equipped with a burner head capable of accepting a solution containing 25 g/L of nickel, as nitrate, and suitable for an air/acetylene flame.

12.1.3 The instrument should be capable of using single-element hollow cathode or electrodeless discharge lamps operated at currents recommended by the instrument manufacturer.

## 13. Reagents

13.1 *Bismuth, Standard Stock Solution* (1 mL = 1 mg Bismuth):

13.1.1 Transfer a 1.00-g sample of bismuth metal (purity, 99.9 % minimum), weighed to the nearest 0.001 g, to a 600-mL beaker.

13.1.2 Add 40 mL of HNO<sub>3</sub> (1 + 1) (~~Note 1~~) and heat gently until dissolution is complete. Boil gently to expel oxides of nitrogen and cool. Transfer to a 1-L volumetric flask containing 160 mL of HNO<sub>3</sub> (1 + 1), dilute to volume with water, and mix. Store in a polyethylene or PTFE bottle. Use the same batch of HNO<sub>3</sub> throughout the entire procedure.

~~Note 1~~—Use the same batch of HNO<sub>3</sub> throughout the entire procedure. Also see ~~Note 4~~.

13.1.3 If inhomogeneity is suspected in the test sample, or if the sample pieces are relatively large, a larger sample mass should be used to prepare the test solution. Under such circumstances, a sample mass of 25 g in a final volume of 1000-mL may be used. The amount of HNO<sub>3</sub> should be increased in proportion. Even larger sample masses can be used, with greater amounts of HNO<sub>3</sub> to prepare a more concentrated nickel test solution. However, an aliquot portion to correspond to a 5-g sample must be taken from such a solution and processed in accordance with the procedure given to give a test solution containing 25 g/L of nickel to match the calibration solutions.

13.2 *Cadmium, Standard Stock Solution* (1 mL = 1 mg Cadmium)—Transfer a 1.00-g sample of cadmium metal (purity, 99.9 % minimum), weighed to the nearest 0.001 g, to a 600-mL beaker. Proceed as directed in 13.1.2.

13.3 *Cobalt, Standard Stock Solution* (1 mL = 1 mg Cobalt)—Transfer a 1.00-g sample of cobalt metal (purity, 99.9 % minimum), weighed to the nearest 0.001 g, to a 600-mL beaker. Proceed as directed in 13.1.2.

13.4 *Copper, Standard Stock Solution* (1 mL = 1 mg Copper)—Transfer a 1.00-g sample of copper metal (purity, 99.9 % minimum), weighed to the nearest 0.001 g, to a 600-mL beaker. Proceed as directed in 13.1.2.

13.5 *Iron, Standard Stock Solution* (1 mL = 1 mg Iron)—Transfer a 1.00-g sample of iron metal (purity, 99.9 % minimum), weighed to the nearest 0.001 g, to a 600-mL beaker. Proceed as directed in 13.1.2.

13.6 *Lead, Standard Stock Solution* (1 mg = 1 mg Lead)—Transfer a 1.00-g sample of lead metal (purity, 99.9 % minimum), weighed to the nearest 0.001 g, to a 600-mL beaker. Proceed as directed in 13.1.2.

13.7 *Manganese, Standard Stock Solution* (1 mL = 1 mg Manganese)—Transfer a 1.00-g sample of manganese metal (purity, 99.9 % minimum), weighed to the nearest 0.001 g, to a 600-mL beaker. Proceed as directed in 13.1.2.

13.8 *Nickel Powder*—High-purity, containing less than 0.0005 % ~~iron~~ and less than 0.0001 % each of silver, bismuth, cadmium, cobalt, copper, manganese, lead, and zinc.

13.9 *Silver, Standard Stock Solution* (1 mL = 1 mg Silver)—Transfer a 1.00-g sample of silver metal (purity, 99.9 % minimum), weighed to the nearest 0.001 g, to a 600-mL beaker. Proceed as directed in 13.1.2, except store in an amber glass container.

13.10 *Zinc, Standard Stock Solution* (1 mL = 1 mg Zinc)—Transfer a 1.00-g sample of zinc metal (purity, 99.9 % minimum), weighed to the nearest 0.001 g, to a 600-mL beaker. Proceed as directed in 13.1.2.

#### 13.11 Working Solutions:

13.11.1 *Mixed Analyte Standard Solution A* (1 mL = 20 µg of silver, bismuth, cadmium, cobalt, copper, iron, manganese, and lead and 10 µg of zinc)—Using pipets, transfer 20.0 mL of each of the standard stock solutions for silver, bismuth, cadmium, cobalt, copper, iron, manganese, and lead and 10 mL of the standard stock solution for zinc to a 1-L volumetric flask containing 160 mL of HNO<sub>3</sub> (1 + 1) (~~1 + 1~~). ~~Use Note 1~~. Use the same batch of HNO<sub>3</sub> throughout the entire procedure. Dilute to volume with water and mix. Store in a glass container.

13.11.2 *Mixed Analyte Standard Solution B* (1 mL = 100 µg of cobalt, copper, iron, and manganese and 10 µg of zinc)—Using pipets, transfer 50.0 mL of the cobalt, copper, iron, and manganese standard stock solutions and 5.0 mL of the zinc standard stock solution to a 500-mL volumetric flask containing 80 mL of HNO<sub>3</sub> (1 + 1). Dilute to volume and mix. Store in a polyethylene or PTFE container.

## 14. Calibration Solutions

### 14.1 Set A:

14.1.1 This set corresponds to (0, 0.2, 0.5, 1.0, 1.5, 2.0, and 2.5) µg/mL each of silver, bismuth, cadmium, cobalt, copper, iron, manganese, and lead and (0, 0.1, 0.25, 0.5, 0.75, 1.0, and 1.25) µg/L of zinc.

Analyte Concentration µ g/mL

No.	Aliquot of Solution A, mL	Silver, Bismuth, Cadmium, Cobalt, Copper, Iron, Manganese, and Lead	Zinc
1	0	0	0
2	2.0	0.2	0.1
3	5.0	0.5	0.25
4	10.0	1.0	0.5
5	15.0	1.5	0.75
6	20.0	2.0	1.0
7	25.0	2.5	1.25

14.1.2 Weigh, to the nearest 0.01 g, seven separate 5.0-g portions of high-purity nickel powder and transfer to 600-mL beakers. Treat as directed in 15.2 to the point of dilution.

14.1.3 Add, using a buret graduated in 0.05-mL divisions, (0, 2.0, 5.0, 10.0, 15.0, 20.0, and 25.0) mL respectively of the mixed Analyte, Standard Solution A to the 200-mL volumetric flasks. Dilute to volume with water and mix. If it is impossible to use the same batch of HNO<sub>3</sub>, a second reagent blank shall be prepared using the same high-purity nickel powder. This blank is then compared with the standard zero calibration solution and an appropriate correction made.

NOTE 2—The solution with zero addition is the reagent blank. See 15.3 and Note 5.

#### 14.2 Set B:

14.2.1 This set corresponds to (0, 2.5, 5.0, 10.0, 15.0, 20.0, and 25.0) µg/mL of cobalt, copper, iron, and manganese and (0, 0.25, 0.5, 1.0, 1.5, 2.0, and 2.5) µg/mL of zinc.

No.	Aliquot of Mixed Analyte, Standard Solution B, mL	Analyte Concentration µg/mL	
		Cobalt, Copper, Iron, and Manganese	Zinc
1	0	0	0
2	5.0	2.5	0.25
3	10.0	5.0	0.5
4	20.0	10.0	1.0
5	30.0	15.0	1.5
6	40.0	20.0	2.0
7	50.0	25.0	2.5

14.2.2 Weigh, to the nearest 0.005 g, seven separate 2.00-g portions of high-purity nickel powder and transfer to 400-mL beakers. Dissolve as directed in 16.2.2.

14.2.3 Using a buret, add (0, 5.0, 10.0, 20.0, 30.0, 40.0, and 50.0) mL respectively of the mixed Analyte, Standard Solution B to the 200-mL volumetric flasks. Dilute to volume with water and mix. The solution with no analyte added is the blank. See Note 5: it is impossible to use the same batch of HNO<sub>3</sub>, a second reagent blank shall be prepared using the same high-purity nickel powder. This blank is then compared with the standard zero calibration solution and an appropriate correction made.

NOTE 3—For convenience, 80 g of nickel/L stock nickel nitrate solution may be prepared by dissolving 20.0 g of nickel powder in water and 120 mL of HNO<sub>3</sub> (1 + 1) in an 800-mL beaker and filtering through acid-washed glass wool or a cellulose filter into a 250-mL volumetric flask. Aliquots (25.0 mL) of this solution are then evaporated and processed as directed in 14.2 and 15.2.

## 15. Procedure A

15.1 This procedure is applicable to 0.0005 % to 0.01 % of silver, bismuth, cadmium, cobalt, copper, iron, manganese, and lead and 0.0005 % to 0.005 % zinc.

15.2 *Preparation of Test Solution*—Weigh, to the nearest 0.01 g, 4.9 g, to 6.1 g of the test sample and transfer to a clean, unetched 600-mL beaker. Add sufficient water to cover the sample and dissolve by adding 60 mL of HNO<sub>3</sub> (1 + 1) in small portions. Heat to complete dissolution, boil gently to expel oxides of nitrogen, and evaporate to a viscous syrup. Redissolve the salts by adding 20 mL HNO<sub>3</sub> (1 + 1) and 100 mL of water. Heat to complete dissolution, cool, and filter, if necessary, through either glass wool or a cellulose filter ~~which has~~ that has been washed with HNO<sub>3</sub> (1 + 1). Collect the filtrate in a 200-mL volumetric flask. Wash the filter with water, collecting the washings, and dilute to volume with water and mix.

NOTE 4—~~If inhomogeneity is suspected in the test sample, or if the sample pieces are relatively large, a larger sample weight should be used to prepare the test solution. Under such circumstances, a sample weight of 25 g in a final volume of 1000-mL may be used. The amount of HNO<sub>3</sub> should be increased in proportion. Even larger sample weights can be used, with greater amounts of HNO<sub>3</sub> to prepare a more concentrated nickel test solution. However, an aliquot portion to correspond to a 5-g sample must be taken from such a solution and processed in accordance with the procedure given to give a test solution containing 25 g/L of nickel to match the calibration solutions.~~

15.2.1 If inhomogeneity is suspected in the test sample, or if the sample pieces are relatively large, a larger sample mass should be used to prepare the test solution. Under such circumstances, a sample mass of 25 g in a final volume of 1000-mL may be used. The amount of HNO<sub>3</sub> should be increased in proportion. Even larger sample masses can be used, with greater amounts of HNO<sub>3</sub> to prepare a more concentrated nickel test solution. However, an aliquot portion to correspond to a 5-g sample must be taken from such a solution and processed in accordance with the procedure given to give a test solution containing 25 g/L of nickel to match the calibration solutions.

15.3 *Reagent Blank Solution*—The zero reference solution of the Calibration Solution Set A (14.1) serves as the reagent blank, since the same batch of HNO<sub>3</sub> is used for dissolution of both the nickel reference and test samples.

NOTE 5—~~If it is impossible to use the same batch of HNO<sub>3</sub>, a second reagent blank shall be prepared using the same high-purity nickel powder. This blank is then compared with the standard zero calibration solution and an appropriate correction made.~~

15.3.1 If it is impossible to use the same batch of HNO<sub>3</sub>, a second reagent blank shall be prepared using the same high-purity nickel powder. This blank is then compared with the standard zero calibration solution and an appropriate correction made.

#### 15.4 Instrumental Parameters:

15.4.1 Use the spectral lines specified in the following table: