



Designation: E1587 – 17

Standard Test Methods for Chemical Analysis of Refined Nickel¹

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 (ϵ) 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	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:

Test Methods	Sections
Antimony, Arsenic, Bismuth, Cadmium, Lead, Selenium, Silver, Tellurium, Tin, and Thallium by the Graphite Furnace Atomic Absorption Spectrometric Method	21 – 31
Bismuth, Cadmium, Cobalt, Copper, Iron, Lead, Manganese, Silver, and Zinc by the Flame Atomic Absorption Spectrometric Method	9 – 20
Sulfur by the Methylene Blue Spectrophotometric Method After Generation of Hydrogen Sulfide	32 – 42

¹ 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.

Current edition approved April 1, 2017. Published June 2017. Originally approved in 1994. Last previous edition approved in 2010 as E1587 – 10. DOI: 10.1520/E1587-17.

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:²

- 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
 - E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry (Withdrawn 2004)³
 - E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
- ### 2.2 ISO Standard:⁴
- ISO 5725 Precision of Test Methods—Determination of Repeatability and Reproducibility by Interlaboratory Tests

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 The last approved version of this historical standard is referenced on www.astm.org.

⁴ 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, 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 sufficiently high purity to permit its use without 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 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](#).

SILVER, BISMUTH, CADMIUM, COBALT, COPPER, IRON, MANGANESE, LEAD, AND ZINC BY FLAME ATOMIC ABSORPTION 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	Mass Fraction Range, %	
	Method A	Method B
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 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 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. For test samples containing greater than 0.25 % and less than 2 % of cobalt or copper, further dilutions of the test solution with HNO₃ (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*

⁵ 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.

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 HNO₃, 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 calibration solutions prepared from high-purity nickel metal. See **Note 1**.

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 HNO₃ 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 HNO₃ (1 + 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 HNO₃ (1 + 1), dilute to volume with water, and mix. Store in a polyethylene or PTFE bottle. Use the same batch of HNO₃ throughout the entire procedure.

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 HNO₃ should be increased in proportion. Even larger sample masses can be used, with greater amounts of HNO₃ 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 HNO₃ (1 + 1). Use the same batch of HNO₃ 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 HNO₃ (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.

No.	Aliquot of Solution A, mL	Analyte Concentration µ g/mL		
		Silver, Bismuth, Cadmium, Cobalt, Copper, Iron, Manganese, and Lead	Zinc	
1	0	0	0	0
2	2.0	0.2	0.1	0.1
3	5.0	0.5	0.25	0.25
4	10.0	1.0	0.5	0.5
5	15.0	1.5	0.75	0.75
6	20.0	2.0	1.0	1.0
7	25.0	2.5	1.25	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 HNO₃, 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.

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 µ g/mL		
		Cobalt, Copper, Iron, and Manganese	Zinc	
1	0	0	0	0
2	5.0	2.5	0.25	0.25
3	10.0	5.0	0.5	0.5
4	20.0	10.0	1.0	1.0
5	30.0	15.0	1.5	1.5
6	40.0	20.0	2.0	2.0
7	50.0	25.0	2.5	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. If it is impossible to use the same batch of HNO₃, 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 HNO₃ (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 HNO₃ (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 HNO₃ (1 + 1) and 100 mL of water. Heat to complete dissolution, cool, and filter, if necessary, through either glass wool or a cellulose filter that has been washed with HNO₃ (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.

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 HNO₃ should be increased in proportion. Even larger sample masses can be used, with greater amounts of HNO₃ 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 HNO₃ is used for dissolution of both the nickel reference and test samples.

15.3.1 If it is impossible to use the same batch of HNO₃, 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:

Spectral Lines—Procedure A					
Element	Silver	Bismuth	Cadmium	Cobalt	Copper
Wavelength, nm	328.1	223.1	228.8	240.7	324.7
Element	Iron	Manganese	Lead	Zinc	
Wavelength, nm	248.3	279.5	217.0	213.9	

15.4.2 The alternative, less-sensitive spectral lines specified in the following table may be used:

Alternate Spectral Lines—Procedure A

Element	Cobalt	Copper	Iron	Manganese	Lead
Wavelength, nm	241.2	327.4	252.3	403.1	283.3

15.4.3 Set the required instrument parameters in accordance with the manufacturer's recommendations. Light the burner and aspirate diluted HNO_3 (1 + 19) until thermal equilibrium is reached. A fuel-lean air-acetylene flame shall be used.

15.4.4 Ensure that the instrument meets the performance requirements given in Practice E60. Optimum settings for the operating parameters vary from instrument to instrument.

15.5 Spectrometry:

15.5.1 Ensure that the test solution (15.2) and the calibration solutions, Set A (14.1) are within 1 °C of the same temperature.

15.5.2 Aspirate diluted HNO_3 (1 + 19) and zero the instrument.

15.5.3 Aspirate the test solution(s) and note the reading to determine its place within the set of calibration solutions.

15.5.4 Aspirate diluted HNO_3 (1 + 19) until the initial reading is obtained. Zero the instrument if necessary.

15.5.5 Aspirate the Set A calibration solutions (14.1) and the test solution(s) in order of increasing instrument response, starting with the zero reference solution. When a stable response is obtained, record the reading. Flush the system by aspirating diluted HNO_3 (1 + 19) between each test or calibration solution. Avoid aspirating the high-nickel solutions for long periods without flushing; otherwise, the burner may tend to clog.

15.5.6 Repeat the measurement of the full set of the calibration and test solutions twice more and record the data. See Note 1.

15.5.7 Proceed with the preparation of the calibration curves and calculations as directed in Sections 17 and 18.

16. Procedure B

16.1 This procedure is applicable to 0.01 % to 0.25 % of cobalt, copper, iron, and manganese and 0.005 % to 0.025 % of zinc.

16.2 Preparation of Test Solution:

16.2.1 If a test solution has been prepared by Procedure A (15.2), using a pipet, transfer a 100.0-mL aliquot portion into a 250-mL volumetric flask, dilute to volume with diluted HNO_3 (1 + 19). Otherwise, proceed as directed in 16.2.2.

16.2.2 Weigh to the nearest 0.005 g, 1.9 g to 2.1 g of the test sample, transfer to a 400-mL beaker and dissolve in 20 mL of HNO_3 (1 + 1). Complete the preparation as directed in 15.2.

16.3 *Reagent Blank Solution*—The zero reference solution of the calibration solution Set B (14.2) serves as the reagent blank. If it is impossible to use the same batch of HNO_3 , 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.

16.4 Instrumental Parameters:

16.4.1 The spectral lines specified in the following table are to be used in the analysis:

Spectral Lines—Procedure B

Element	Cobalt	Copper	Iron	Manganese	Zinc
Wavelength, nm	241.2	327.4	252.3	403.1	213.9

16.4.2 Proceed as directed in 15.4.3 and 15.4.4.

16.5 Spectrometry:

16.5.1 Proceed as directed in 15.5.1 through 15.5.6, substituting the Set B calibration solution (14.2) for the Set A solutions.

16.5.2 Proceed with the preparation of the calibrations curves and calculations as directed in Sections 17 and 18.

17. Preparation of Calibration Curves

17.1 Plot the average instrument reading against the concentration of the analyte for the calibration solutions for each set of measurements.

17.2 For instruments that have automated calibration features and direct read-out in concentration, plotting of calibration curves is not required. Follow the instrument operating instructions for calibration and curvature correction procedures.

18. Calculations

18.1 Determine the concentration of analyte in the test solution from the corresponding calibration curves or instrument read-out for each of the three sets of instrument readings. Average the resultant concentrations.

18.2 *Procedure A*—Calculate the mass fraction of the analyte in the test sample as follows:

$$\text{Analyte, \%} = \frac{A \times B}{C} \times 10^{-4} \quad (1)$$

where:

A = analyte concentration found in the test solution, $\mu\text{g/mL}$,

B = volume of the test solution, mL, and

C = mass of the test sample, g.

18.3 Procedure B:

18.3.1 For the procedure in 16.2.1, calculate mass fraction of the analyte in the test sample as follows:

$$\text{Analyte, \%} = \frac{A \times B}{C} \times 2.5 \times 10^{-4} \quad (2)$$

where 2.5 = correction factor for the dilution made.

19. Precision and Bias

19.1 Precision:

19.1.1 Eighteen laboratories in nine countries participated in testing this method under the auspices of ISO/TC-155/SC-3/WG-1 in the early 1980s and obtained the statistical data summarized in Table 1 as evaluated by ISO 5725 and equivalent to Practice E1601. Precision may be judged by examination of these data. Twelve sample were analyzed to cover the scope of this test method. Of these, ten were specially prepared as no materials containing the impurity levels were available commercially.

19.1.2 The laboratory test program was designed so that the statistics on repeatability would include variations because of a

TABLE 1 Statistical Information—Flame AAS Method, Procedure A

Test Material	Mean, %	Repeatability Index, <i>r</i> (Practice E1601)	Reproducibility, Index <i>R</i> (Practice E1601)
Silver			
P45	0.00043	0.00003	0.00012
P44	0.00077	0.00005	0.00007
P46	0.00095	0.00012	0.00015
P41	0.00191	0.00008	0.00017
J63	0.00232	0.00010	0.00022
P43	0.00282	0.00017	0.00022
J61	0.00970	0.00025	0.00142
Bismuth			
P44	0.00133	0.00027	0.00076
P41	0.00171	0.00028	0.00047
P43	0.00245	0.00031	0.00049
J61	0.01037	0.00044	0.00057
Cadmium			
P46	0.00019	0.00003	0.00008
J63	0.00025	0.00002	0.00009
J61	0.00135	0.00007	0.00025
S65	0.00225	0.00007	0.00025
Cobalt			
P43	0.00105	0.00007	0.00016
P44	0.00155	0.00007	0.00040
P41	0.00185	0.00011	0.00014
J62	0.00508	0.00023	0.00030
J61	0.01002	0.00038	0.00060
Copper			
S65	0.00079	0.00012	0.00022
J62	0.00517	0.00009	0.00025
J61	0.01006	0.00009	0.00041
Iron			
P46	0.00241	0.00020	0.00059
P45	0.00298	0.00033	0.00060
P44	0.00311	0.00013	0.00058
P41	0.00437	0.00018	0.00103
S65	0.00474	0.00026	0.00058
Manganese			
P41	0.00054	0.00003	0.00020
P46	0.00070	0.00005	0.00020
P45	0.00107	0.00008	0.00020
P43	0.00200	0.00005	0.00014
J62	0.00536	0.00013	0.00037
J61	0.01028	0.00027	0.00052
Lead			
H79	0.00078	0.00003	0.00017
P46	0.00090	0.00030	0.00036
P41	0.00202	0.00032	0.00048
P44	0.00252	0.00024	0.00026
J62	0.00350	0.00011	0.00041
J63	0.00365	0.00017	0.00017
J61	0.00777	0.00020	0.00046
Zinc			
H79	0.00029	0.00004	0.00015
P44	0.00041	0.00004	0.00020
P41	0.00050	0.00007	0.00016
P46	0.00062	0.00008	0.00010
S65	0.00101	0.00009	0.00017
P43	0.00117	0.00009	0.00028
P45	0.00128	0.00023	0.00040
J62	0.00269	0.00014	0.00024

if the nickel metal used for the preparation of the calibration solutions does not meet the purity specifications given in the test method and appropriate corrections are not made.

20. Keywords

20.1 bismuth; cadmium; cobalt; copper; flame atomic absorption spectrometry; iron; lead; manganese; refined nickel; silver; spectrometry; zinc

SILVER, ARSENIC, BISMUTH, CADMIUM, LEAD, ANTIMONY, SELENIUM, TIN, TELLURIUM, AND THALLIUM BY THE GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY

21. Scope

21.1 This test method applies to the determination of the silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium, and thallium contents of high-purity, refined, wrought, and cast nickel metal within the ranges specified in the following table:

Element	Mass Fraction Range, µg/g
Silver	0.3 to 10
Arsenic	1.3 to 20
Bismuth	4.0 to 15
Cadmium	0.3 to 2
Lead	0.7 to 10
Antimony	1.8 to 10
Selenium	1.8 to 10
Tin	2.2 to 5
Tellurium	1.5 to 10
Thallium	0.5 to 10

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

21.3 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*.

22. Summary of Test Method

22.1 The test sample is dissolved in HNO₃ and the solution is diluted to a known volume. An aliquot is introduced into a graphite furnace atomic absorption spectrometer (GF-AAS) and 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. All readings are background-corrected.

23. Interferences

23.1 Elements ordinarily present in nickel metal do not present spectral interferences in graphite furnace atomic absorption analysis.

23.2 Potential background absorption interference is eliminated by instrumental background correction and by the use of matched-matrix calibration solutions prepared from high-purity nickel metal.

change in the atomic absorption instrument or operator, or both, while maintaining the same test solution.

19.2 *Bias*—No information is currently available on the bias of this test method, because of the lack of appropriate certified reference materials. The bias of a test method may be judged, however, by comparing accepted reference values with the arithmetic average obtained by interlaboratory testing. The user is cautioned that the results will be biased to the low side