



Standard Test Method for Analysis of Nickel Alloys by Flame Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation E1835; 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 analysis of nickel alloys by flame atomic absorption spectrometry (FAAS) for the following elements:

Element	Composition Range, %
Aluminum	0.2 to 4.0
Chromium	0.01 to 4.0
Cobalt	0.01 to 4.0
Copper	0.01 to 4.0
Iron	0.1 to 4.0
Manganese	0.1 to 4.0
Silicon	0.2 to 1.0
Vanadium	0.05 to 1.0

1.2 The composition ranges of these elements can be expanded by the use of appropriate standards.

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 and the warning statements included in this test method.

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 Consid-

¹ This test method 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.

erations 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

E1812 Practice for Optimization of Flame Atomic Absorption Spectrometric Equipment (Withdrawn 2004)³

2.2 *ISO Standards:*⁴

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

ISO 7530 Parts 1 through 9—Nickel Alloys—Flame Atomic Absorption Spectrometric Analysis

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 HNO₃. The solution is aspirated into an appropriate flame of an atomic absorption spectrometer. The absorbance of the resonant line energy from the spectrum of the analyte is measured and compared with that of calibration solutions.

5. Significance and Use

5.1 This test method is used for the analysis of nickel alloy samples by FAAS 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.

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.

TABLE 1 Nominal Compositions of Test Samples, %

Test Material	Al	Co	Cr	Cu	Fe	Mn	Mo	Nb	Ni	Si	Ti	V	Zr
825	0.2	0.07	21	1.6	30	0.7	Bal	0.4	1.1
902	0.4	0.05	5	0.04	48	0.4	Bal	0.35	2.5
3920	0.15	2	19	0.1	3	0.3	Bal	0.6	2.3
3927	0.1	1	20	0.05	44	0.4	Bal	0.8	0.6
7013	1.5	17	20	0.2	0.2	0.05	Bal	0.7	2.4
7049	1	0.01	15	0.15	7	0.8	Bal	0.3	2.3
925	0.3	0.2	21	...	27	...	3	0.4	Bal	...	2	0.05	0.05
NPK31	0.5	14	20	...	1	...	4.5	5	Bal	...	2	0.3	...
IN100	5.5	15	10	...	<0.5	...	3	...	Bal	...	5	1	...

5.2 *Interlaboratory Studies (ILS)*^{5, 6}—International interlaboratory studies were conducted by ISO/TC 155/SC4, Analysis of nickel alloys. Results were evaluated in accordance with ISO 5725:1986 and restated to conform to Practice E1601. The method was published as ISO 7530, Parts 1 through 9. The published ISO statistics are summarized separately for each analyte to correspond with Practice E1601.

5.3 In this test method, some matrix modifiers are specified. However, other additives have come into common use since the original publication of this test method. These may be equally or more effective but have not been tested. It is the responsibility of the user to validate the use of such additives or the use of different dilutions, or both.

6. Apparatus

6.1 *Flame Atomic Absorption Spectrometer*, equipped with an appropriate background corrector, a signal output device (such as a video display screen (VDS), a digital computer, a printer or strip chart recorder, and an optional autosampler.

6.2 *Radiation Source*—Hollow cathode lamp or electrodeless discharge lamp for the analyte(s).

7. Reagents

7.1 *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.⁷ 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. The reagents should be free of or contain minimal amounts (<0.1 µg/g) of the analyte of interest.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or II of Specification D1193. Type III or IV may be used if they effect no measurable change in the blank or sample.

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report : RR:E01-1018.

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report : RR:E01-1019.

⁷ *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.

7.3 *Calibration Solutions*—Prepared for the individual analytes.

7.4 *Matrix Modifiers and Ionization Buffers*—Prepared for the individual analytes, where required.

8. Sampling and Sample Preparation

8.1 Sampling and sample preparation 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 sampling procedure shall not involve any steps or procedures that can result in the loss of any analyte in the sample.

NOTE 1—Arc melting of the sample or induction melting of the sample under vacuum can result in significant loss of several elements that have a low vapor pressure. Arc melting of the sample should be performed only after careful consideration of all elements to be determined on the melted sample. Induction melting should be performed only in a complete or partial inert atmosphere.

8.3 The laboratory sample is normally in the form of turnings, millings, or drillings and no further mechanical preparation is necessary.

8.4 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.

8.5 If brazed alloy tools have been used in the preparation of the sample, it shall be further cleaned by pickling in dilute HNO₃ for a few minutes. The sample shall then be washed several times with water followed by several washes with high purity acetone, or other appropriate solvent, and dried in air.

9. General Procedure

9.1 Sample Dissolution:

9.1.1 Transfer a 1.0-g sample, weighed to the nearest 1 mg, to a 600-mL beaker. Add 15 mL HCl and 5 mL HNO₃. Apply sufficient heat to initiate and maintain the reaction until the dissolution is complete. If the sample contains over 0.5 % silicon, a few drops of HF will speed up the dissolution considerably. (**Warning**—This operation will emit corrosive, noxious, and toxic gases and should only be performed in a fume hood. Proper personal safety equipment shall be worn and used.)

9.1.2 If the sample resists dissolution, some adjustment of the acid mixture may be required. Add HCl in 1-mL increments and continue heating to dissolve the sample.

NOTE 2—For some alloys a 30-mL HCl—2-mL HNO₃ mixture is more effective. Nickel alloys dissolve best in HNO₃ (1 + 1).

NOTE 3—The general method of dissolution may be modified as specified in the appropriate sections.

NOTE 4—If sample inhomogeneity is suspected, a larger mass of sample (10 g to 50 g) may be taken for analysis. However, an aliquot portion corresponding to 1-g sample shall be taken from the solution and processed in accordance with the procedure given.

9.1.3 Using low heat, evaporate the solution just to dryness. Do not bake. Cool to about 50 °C and add 25 mL HCl and again evaporate just to dryness. Add 25 mL HCl and repeat the evaporation.

9.1.4 Cool to about 50 °C, add 5 mL HCl and 20 mL water and heat to dissolve the salt.

9.1.5 Proceed as directed in Sections 12 through 19.

9.2 *Reagent Blank*—Carry a reagent blank through the entire procedure using the same amounts of all reagents with the sample omitted.

9.3 *Calibration Solutions*—Proceed as directed in Sections 12 through 19.

9.4 *Atomic Absorption Measurements:*

9.4.1 The wavelengths of the spectral lines and the flame types to be used are listed in Sections 12 through 19.

9.4.2 Set the required instrument parameters in accordance with the manufacturer's recommendations or Practice E1812. Light the burner and aspirate water until thermal equilibrium is reached. The flame conditions will vary according to the element being determined. Zero the instrument.

9.4.3 Ensure that the instrument meets the performance requirements given in Practice E1812. Optimum settings for the operating parameters vary from instrument to instrument. Scale expansion may have to be used to obtain the required readability.

9.4.4 Ensure that the calibration solutions and the test solution(s) are within 1 °C of the same temperature.

9.4.5 Aspirate water and zero the instrument.

9.4.6 Aspirate the calibration solutions and the test solution(s) and note the readings to determine the approximate concentration of the test solution(s).

9.4.7 Aspirate water until the initial reading is obtained. Zero if necessary.

9.4.8 Aspirate the calibration solutions and the test solution(s) in the order of increasing instrument response, starting with the calibration solution containing no analyte (*S*₀). When a stable response is obtained record the reading. Flush the system by aspirating water between each test and calibration solution.

9.4.9 Repeat the measurement of the full set of calibration and test solutions two more times and record the data.

10. Preparation of Calibration Graphs

10.1 For each calibration solution, calculate the average of the replicate absorbance measurements made in 9.4.9. Then, plot the average absorbance values versus the concentrations of the analyte in the calibration solutions.

NOTE 5—Since the testing of these methods, there have been many advances in instrument technology for FAAS and the procedures for calibration, making the manual plotting of calibration graphs redundant.

10.2 Conduct measurements at least in triplicate.

11. Calculation

11.1 Determine the concentration of the analyte in the test solution from the corresponding calibration graphs for each of the three sets of instrument readings recorded.

11.2 Calculate the percentage of the analyte in the test sample using the formula:

$$\text{Analyte, \%} = (c V F) / 10000 m \quad (1)$$

where:

c = analyte concentration, mg/L, found in the test solution, less the blank;

V = volume, mL, of the initial test solution;

F = dilution factor for the secondary dilution; and

m = mass, g, of the test portion.

11.3 Rounding of test results obtained using this test method shall be performed in accordance with Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification.

12. Determination of Aluminum

12.1 *Parameters:*

12.1.1 *Wavelength:* 309.3 nm.

12.1.2 *Flame:* nitrous oxide—Acetylene.

12.2 *Reagents:*

12.2.1 *Potassium Chloride Ionization Buffer Solution* (48 g/L)—Dissolve 48 g potassium chloride (KCl) in 500 mL of water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

12.2.2 *Aluminum Stock Calibration Solution* (1.00 g/L)—Dissolve 1.00 g of aluminum (purity 99.99 % min) in 30 mL of HCl (1 + 1). (**Warning**—If powdered aluminum is used, add the acid cautiously because powdered aluminum tends to be very reactive). Place the beaker on a hot plate and heat the solution to approximately 90 °C to start the reaction. Remove the beaker from the hotplate when the reaction starts and cover with a watch glass. Pure aluminum dissolves slowly in HCl and complete dissolution may take several days. After complete dissolution add 1 mL of 30 % H₂O₂ and place the beaker on a hotplate. Heat the solution to about 110 °C and gently boil for about 5 min. Cool and transfer to a 1000 mL volumetric flask. Add 85 mL of HCl to the flask, dilute to volume with water, and mix well. Store in a polycarbonate container.

12.2.3 *Aluminum Calibration Solution* (100 mg/L)—Transfer a 100-mL aliquot of the aluminum stock standard solution (12.2.2) into a 1-L volumetric flask. Add 90 mL of HCl and 800 mL water. Cool, dilute to volume, and mix. Store in a polyethylene bottle.

12.3 *Aluminum Calibration Solutions*—Transfer to each of six 100-mL volumetric flasks (0, 5.0, 10.0, 15.0, 20.0, and 25.0) mL, respectively, of the aluminum calibration solution (12.2.3). Add 4 mL of the KCl solution and 4 mL of HNO₃ to each volumetric flask. Add (10.0, 9.5, 9.0, 8.5, 8.0, and 7.5) mL of HCl, respectively, to the six volumetric flasks. Cool, dilute to volume, and mix. The calibration solutions are identified as

S₀ through S₅ and contain (0, 5.0, 10.0, 15.0, 20.0, and 25.0) mg/L Al, respectively.

NOTE 6—It is important that all calibration solutions contain the same amount (10 % v/v) of HCl, including the 10 % HCl contained in the aluminum calibration solution (12.2.3).

12.4 Sample Dissolution and Dilution:

12.4.1 Transfer a 1-g sample, weighed to the nearest 1 mg, to a 400-mL PTFE beaker and add 15 mL of HCl and 5 mL of HNO₃. Heat to initiate and maintain the reaction until dissolution is complete. If any alloy resists dissolution, add HCl in 1-mL increments and continue to heat to dissolve sample.

12.4.2 Dilute the solution to 50 mL with water and filter through 11-cm low-ash medium-porosity filter paper into a 250-mL beaker. Wash the filter five times with 10-mL portions of hot water. Add the washings to the filtrate. Reserve the filter paper containing any undissolved residue.

12.4.3 *Primary Dilutions for Samples Containing Less Than 0.25 % Aluminum*—Evaporate the filtrate reserved from 12.4.2 to approximately 60 mL. Cool and transfer to a 100-mL volumetric flask. Add 2.5 mL HCl, 4 mL HNO₃, and 4 mL KCl solution. Cool, dilute to volume, and mix.

12.4.4 *Primary Dilution for Samples Containing Over 0.25 % Aluminum*—Evaporate the filtrate reserved from 12.4.2 to approximately 60 mL. Cool and transfer to a 100-mL volumetric flask. Add 2.5 mL HCl, dilute to volume, and mix.

12.4.5 *Secondary Dilution for Samples Containing Between 0.25 % and 1.0 % Aluminum*—Transfer 20 mL of the primary dilution solution (12.4.4) into a 100-mL volumetric flask, and add 8 mL of HCl, 4 mL of HNO₃, and 4 mL of KCl solution. Cool, dilute to mark, and mix. The dilution factor F = 5.

12.4.6 *Secondary Dilution for Samples Containing Between 1.0 % and 2.0 % Aluminum*—Transfer 10 mL of the primary dilution solution (12.4.4) into a 100-mL volumetric flask, and add 9 mL of HCl, 4 mL of HNO₃, and 4 mL of KCl solution. Cool, dilute to mark, and mix. The dilution factor F = 10.

12.4.7 *Secondary Dilution for Samples Containing Between 2.0 % and 4.0 % Aluminum*—Transfer 5 mL of the primary dilution solution (12.4.4) into a 100-mL volumetric flask and add 9.5 mL of HCl, 4 mL of HNO₃, and 4 mL of KCl solution. Cool, dilute to mark, and mix. The dilution factor F = 20.

12.4.8 Transfer the reserved filter containing any undissolved residue from 12.4.2 to a platinum crucible. Dry, char, and ignite to oxidize the carbon and cool. Add 0.25 mL (1 + 1) H₂SO₄ and 1 mL HF. Carefully evaporate to dryness and fuse residue with 1 g of potassium pyrosulfate. Allow the melt to cool and dissolve in a small volume of water containing 0.25 mL of HCl. Heat, if necessary, to complete dissolution.

TABLE 2 Results of Statistical Analysis—Aluminum

Test Material ^A	Mean, %	Repeatability Index r (Practice E1601)	Reproducibility Index R (Practice E1601)
3927	0.109	0.0091	0.016
3920	0.146	0.0047	0.012
825	0.169	0.015	0.035
902	0.434	0.013	0.025
7049	0.972	0.025	0.035
7013	1.51	0.034	0.037

^A Nominal material compositions are summarized in Table 1.

TABLE 3 Results of Statistical Analysis—Chromium

Test Material ^A	Mean, %	Repeatability Index r (Practice E1601)	Reproducibility Index R (Practice E1601)
902	5.16	0.096	0.30

^A Nominal material compositions are summarized in Table 1.

12.4.9 Transfer the leach solution to a 100-mL volumetric flask and add 10 mL HCl. Dilute with 25 mL of water and add 4 mL of HNO₃. Cool and dilute to volume and mix.

NOTE 7—A very small amount of aluminum may be present in the fused residue, but it usually does not exceed 0.5 mg. The solution is analyzed separately and the aluminum found is added to the main result.

12.5 *Calibration, Determination, and Calculation*—Complete the calibration, determination, and calculation in accordance with Section 11.

12.6 Precision and Bias:^{5,6}

12.6.1 *Precision*—Six laboratories in four countries cooperated in testing this method and obtained statistical information summarized in Table 2.

12.6.2 *Bias*—No information on the accuracy of this method is known because accepted reference standards were not used in the ILS. The user of the method is encouraged to use accepted reference materials, if available, to determine the accuracy of this method as applied in a specific laboratory.

13. Determination of Chromium

13.1 Parameters:

13.1.1 *Wavelength*: 357.9 nm.

13.1.2 *Flame*: nitrous oxide—Acetylene.

13.2 Reagents:

13.2.1 *Strontium Chloride Ionization Buffer Solution*—Dissolve 113.5 g of strontium chloride hexahydrate (SrCl₂ · 6H₂O) in 400 mL of hot water (50 °C to 60 °C). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

13.2.2 *Chromium Stock Calibration Solution* (1.000 g/L)—Dissolve 1.000 g of chromium (purity 99.9 % min) in 30 mL HCl (1 + 1). Heat to complete dissolution. Cool, transfer to a 1-L volumetric flask, add 35 mL of HCl, dilute to volume, and mix. Store in a high-density polyethylene bottle.

13.2.3 *Chromium Calibration Solution* (50 mg/L)—Transfer 50 mL of the chromium stock calibration solution (13.2.2) into a 1-L volumetric flask and add 50 mL of HCl. Dilute to volume and mix. Store in a high-density polyethylene bottle.

13.3 *Chromium Calibration Solutions*—Transfer to each of five 100-mL volumetric flasks (0, 5.0, 10.0, 15.0, and 20.0) mL, respectively, of the chromium calibration solution (13.2.3). Add 4 mL of the SrCl₂ solution and 5 mL of HCl to each volumetric flask. Dilute to volume and mix. The calibration solutions are identified as S₀ through S₅ and contain (0, 2.5, 5.0, 7.5, and 10.0) mg/L of Cr, respectively.

13.4 Sample Dissolution and Dilution:

13.4.1 Dissolve samples in accordance with 9.1 – 9.1.4.

13.4.2 *Primary Dilution for Samples Containing Less Than 0.10 % Chromium*—Transfer the dissolved sample to a 100-mL volumetric flask. Add 4 mL of SrCl₂ solution, cool, dilute to

volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

13.4.3 *Primary Dilution for Samples Containing More Than 0.10 % Chromium*—Transfer the dissolved sample to a 500-mL volumetric flask, add 20 mL of HCl, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

13.4.4 *Secondary Dilution for Samples Containing Between 0.1 % and 0.8 % Chromium*—Transfer 50 mL of the primary dilution solution (13.4.3) into a 100-mL volumetric flask and add 4 mL of SrCl₂ and 3 mL of HCl. Cool, dilute to mark, and mix. The dilution factor F = 2.

13.4.5 *Secondary Dilution for Samples Containing Between 0.8 % and 4.0 % Chromium*—Transfer 10 mL of the primary dilution solution (13.4.3) into a 100-mL volumetric flask and add 4 mL of SrCl₂ and 5 mL of HCl. Cool, dilute to mark, and mix. The dilution factor F = 10.

13.5 *Calibration, Determination, and Calculation*—Complete the calibration, determination, and calculation in accordance with 9.2 through Section 11.

13.6 *Precision and Bias*:^{5,6}

13.6.1 *Precision*—Ten laboratories in five countries cooperated in testing this method and obtained statistical information summarized in Table 3.

13.6.2 *Bias*—No information on the accuracy of this method is known because accepted reference standards were not used in the ILS. The user of the method is encouraged to use accepted reference materials, if available, to determine the accuracy of this method as applied in a specific laboratory.

14. Determination of Cobalt

14.1 *Parameters*:

14.1.1 *Wavelength*: 240.7 nm.

14.1.2 *Flame*: air—Acetylene.

14.2 *Reagents*:

14.2.1 *Strontium Chloride Ionization Buffer Solution*—Dissolve 113.5 g of SrCl₂ · 6H₂O in 400 mL of hot water (50 °C to 60 °C). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

14.2.2 *Cobalt Stock Calibration Solution (1.000 g/L)*—Dissolve 1.000 g of cobalt (purity 99.9 % min) in 30 mL HCl (1 + 1). Heat to complete dissolution. Cool, transfer to a 1-L volumetric flask, add 35 mL of HCl, dilute to volume, and mix. Store in a high-density polyethylene bottle.

14.2.3 *Cobalt Calibration Solution (50 mg/L)*—Transfer 50 mL of the cobalt stock calibration solution (14.2.2) into a 1-L volumetric flask and add 50 mL of HCl. Dilute to volume and mix. Store in a high-density polyethylene bottle.

14.3 *Cobalt Calibration Solutions*—Transfer to each of five 100-mL volumetric flasks (0, 5.0, 10.0, 15.0, and 20.0) mL, respectively, of the cobalt calibration solution (14.2.3). Add 4 mL of the SrCl₂ solution and 5 mL of HCl to each volumetric flask. Dilute to volume and mix. The calibration solutions are identified as S₀ through S₄ and contain (0, 2.5, 5.0, 7.5, and 10.0) mg/L Co, respectively.

14.4 *Sample Dissolution and Dilution*:

14.4.1 Dissolve samples in accordance with 9.1 – 9.1.4.

14.4.2 *Primary Dilution for Samples Containing Less Than 0.10 % Cobalt*—Transfer the dissolved sample to a 100-mL volumetric flask. Add 4 mL of SrCl₂ solution, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

14.4.3 *Primary Dilution for Samples Containing More Than 0.10 % Cobalt*—Transfer the dissolved sample to a 500-mL volumetric flask, add 20 mL of HCl, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

14.4.4 *Secondary Dilution for Samples Containing Between 0.1 % and 0.8 % Cobalt*—Transfer 50 mL of the primary dilution solution (14.4.3) into a 100-mL volumetric flask and add 4 mL of SrCl₂ solution and 3 mL of HCl. Cool, dilute to mark, and mix. The dilution factor F = 2.

14.4.5 *Secondary Dilution for Samples Containing Between 0.8 % and 4.0 % Cobalt*—Transfer 10 mL of the primary dilution solution (14.4.3) into a 100-mL volumetric flask and add 4 mL of SrCl₂ solution and 5 mL of HCl. Cool, dilute to mark, and mix. The dilution factor F = 10.

14.5 *Calibration, Determination, and Calculation*—To complete the calibration, determination, and calculation in accordance with through Section 11.

14.6 *Precision and Bias*:^{5,6}

14.6.1 *Precision*—Twelve laboratories in six countries cooperated in testing this method and obtained statistical information summarized in Table 4.

14.6.2 *Bias*—No information on the accuracy of this method is known because accepted reference standards were not used in the ILS. The user of the method is encouraged to use accepted reference materials, if available, to determine the accuracy of this method as applied in a specific laboratory.

15. Determination of Copper

15.1 *Parameters*:

15.1.1 *Wavelength*: 324.8 nm.

15.1.2 *Flame*: air—Acetylene.

15.2 *Reagents*:

15.2.1 *Strontium Chloride Ionization Buffer Solution*—Dissolve 113.5 g of SrCl₂ · 6H₂O in 400 mL of hot water (50 °C to 60 °C). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

15.2.2 *Copper Stock Calibration Solution (1.000 g/L)*—Dissolve 1.000 g copper (purity 99.9 % min) in 50 mL HNO₃ (1 + 1). Allow to stand until reaction ceases. Heat to complete dissolution and boil to remove oxides of nitrogen. Evaporate just to dryness. Cool, and add 25 mL of HCl and evaporate just to dryness. Add another 25 mL of HCl and repeat evaporation.

TABLE 4 Results of Statistical Analysis—Cobalt

Test Material ^A	Mean, %	Repeatability Index r (Practice E1601)	Reproducibility Index R (Practice E1601)
7049	0.0072	0.0006	0.0026
902	0.046	0.0035	0.0060
825	0.067	0.0016	0.0060
3927	1.03	0.045	0.077
3920	2.01	0.082	0.082

^A Nominal material compositions are summarized in Table 1.

Dissolve salts in 50 mL HCl (1 + 1), heat if necessary. Cool, transfer to a 1-L volumetric flask, add 35 mL HCl, dilute to volume, and mix. Store in a high-density polyethylene bottle.

15.2.3 *Copper Calibration Solution* (50 mg/L)—Transfer 50 mL of the copper stock calibration solution into a 1-L volumetric flask and add 50 mL of HCl. Dilute to volume and mix. Store in a high-density polyethylene bottle.

15.3 *Copper Calibration Solutions*—Transfer to each of five 100-mL volumetric flasks (0, 5.0, 10, 15, and 20) mL, respectively, of the copper calibration solution. Add 4 mL of the SrCl₂ solution and 5 mL of HCl to each flask. Dilute to volume and mix. The calibration solutions are identified as S₀ through S₄ and contain (0, 2.5, 5.0, 7.5, and 10.0) mg/L Cu, respectively.

15.4 *Sample Dissolution and Dilution:*

15.4.1 Dissolve samples in accordance with 9.1 – 9.1.4.

15.4.2 *Primary Dilution for Samples Containing Less Than 0.10 % Copper*—Transfer the dissolved sample to a 100-mL volumetric flask. Add 4 mL of SrCl₂ solution, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

15.4.3 *Primary Dilution for Samples Containing More Than 0.10 % Copper*—Transfer the dissolved sample to a 500-mL volumetric flask, add 20 mL of HCl, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

15.4.4 *Secondary Dilution for Samples Containing Between 0.1 % and 0.8 % Copper*—Transfer 50 mL of the primary dilution solution into a 100-mL volumetric flask, and add 4 mL of SrCl₂ solution and 3 mL of HCl. Cool, dilute to mark, and mix. The dilution factor F = 2.

15.4.5 *Secondary Dilution for Samples Containing Between 0.8 % and 4.0 % Copper*—Transfer 10 mL of the primary dilution solution into a 100-mL volumetric flask and add 4 mL of SrCl₂ solution and 5 mL of HCl. Cool, dilute to mark, and mix. The dilution factor F = 10.

15.5 *Calibration, Determination, and Calculation*—Complete the calibration, determination, and calculation in accordance with 9.2 through Section 11.

15.6 *Precision and Bias:*^{5, 6}

15.6.1 *Precision*—Twelve laboratories in six countries cooperated in testing this method and obtained statistical information summarized in Table 5.

15.6.2 *Bias*—No information on the accuracy of this method is known because accepted reference standards were not used in the ILS. The user of the method is encouraged to use

TABLE 5 Results of Statistical Analysis—Copper

Test Material ^A	Mean, %	Repeatability Index r (Practice E1601)	Reproducibility Index R (Practice E1601)
902	0.036	0.0012	0.0035
3927	0.054	0.0024	0.0066
3920	0.11	0.008	0.016
7049	0.16	0.008	0.017
7013	0.20	0.006	0.013
825	162	0.016	0.053

^A Nominal material compositions are summarized in Table 1.

TABLE 6 Results of Statistical Analysis—Iron

Test Material ^A	Mean, %	Repeatability Index r (Practice E1601)	Reproducibility Index R (Practice E1601)
7013	0.21	0.007	0.019
3920	3.00	0.083	0.16

^A Nominal material compositions are summarized in Table 1.

accepted reference materials, if available, to determine the accuracy of this method as applied in a specific laboratory.

16. Determination of Iron

16.1 *Parameters:*

16.1.1 *Wavelength:* 248.3 nm.

16.1.2 *Flame:* air—Acetylene.

16.2 *Reagents:*

16.2.1 *Strontium Chloride Ionization Buffer Solution*—Dissolve 113.5 g of SrCl₂ · 6H₂O in 400 mL of hot water (50 °C to 60 °C). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

16.2.2 *Iron Stock Calibration Solution* (1.000 g/L)—Dissolve 1.000 g of iron (purity 99.9 % min) in 30 mL HCl (1 + 1). Heat to initiate the reaction and complete dissolution. Cool to about 50 °C, cautiously add 1 mL hydrogen peroxide (30 %), and bring to a boil to oxidize the iron. Cool, transfer to a 1-L volumetric flask, add 35 mL of HCl, dilute to volume, and mix. Store in a high-density polyethylene bottle.

16.2.3 *Iron Calibration Solution* (50 mg/L)—Transfer 50 mL of the iron stock calibration solution into a 1-L volumetric flask and add 50 mL of HCl. Dilute to volume and mix. Store in a high-density polyethylene bottle.

16.3 *Iron Calibration Solutions*—Transfer to each of five 100-mL volumetric flasks (0, 5.0, 10.0, 15.0, and 20.0) mL, respectively, of the iron calibration solution. Add 4 mL of the SrCl₂ solution and 5 mL of HCl to each flask. Dilute to volume and mix. The calibration solutions are identified as S₀ through S₄ and contain (0, 2.5, 5.0, 7.5, and 10.0) mg/L of Fe, respectively.

16.4 *Sample Dissolution and Dilution:*

16.4.1 Dissolve samples in accordance with 9.1 – 9.1.4.

16.4.2 *Primary Dilution for Samples Containing Less Than 0.10 % Iron*—Transfer the dissolved sample to a 100-mL volumetric flask. Add 4 mL of SrCl₂ solution, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

16.4.3 *Primary Dilution for Samples Containing More Than 0.10 % Iron*—Transfer the dissolved sample to a 500-mL volumetric flask, add 20 mL of HCl, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

16.4.4 *Secondary Dilution for Samples Containing Between 0.1 % and 0.8 % Iron*—Transfer 50 mL of the primary dilution solution into a 100-mL volumetric flask and add 4 mL of SrCl₂ solution and 3 mL of HCl. Cool, dilute to mark, and mix. The dilution factor F = 2.

16.4.5 *Secondary Dilution for Samples Containing Between 0.8 % and 4.0 % Iron*—Transfer 10 mL of the primary dilution solution into a 100-mL volumetric flask and add 4 mL of SrCl₂

solution and 5 mL of HCl. Cool, dilute to mark, and mix. The dilution factor $F = 10$.

16.5 Calibration, Determination, and Calculation—Complete the calibration, determination, and calculation in accordance with 9.2 through Section 11.

16.6 Precision and Bias:^{5,6}

16.6.1 Precision—Eleven laboratories in six countries cooperated in testing this method and obtained statistical information summarized in Table 6.

16.6.2 Bias—No information on the accuracy of this method is known because accepted reference standards were not used in the ILS. The user of the method is encouraged to use accepted reference materials, if available, to determine the accuracy of this method as applied in a specific laboratory.

17. Determination of Manganese

17.1 Parameters:

17.1.1 Wavelength—279.5 nm.

17.1.2 Flame: air—Acetylene.

17.2 Reagents:

17.2.1 Strontium Chloride Ionization Buffer Solution—Dissolve 113.5 g of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ in 400 mL of hot water (50 °C to 60 °C). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

17.2.2 Manganese Stock Calibration Solution (1.000 g/L)—Dissolve 1.000 g of manganese (purity 99.9 % min) in 30 mL HCl (1 + 1) and 2 mL of HNO_3 . Heat to initiate the reaction and complete dissolution. Cool to about 50 °C, cautiously add 0.5 mL of hydrogen peroxide (30 %). Cool, transfer to a 1-L volumetric flask, add 50 mL HCl, dilute to volume, and mix. Store in a high-density polyethylene bottle.

17.2.3 Manganese Calibration Solution (50 mg/L)—Transfer 50 mL of the manganese stock calibration solution into a 1-L volumetric flask and add 50 mL of HCl. Dilute to volume and mix. Store in a high-density polyethylene bottle.

17.3 Manganese Calibration Solutions—Transfer to each of five 100-mL volumetric flasks (0, 5.0, 10.0, 15.0, and 20.0) mL, respectively, of the manganese calibration solution. Add 4 mL of the SrCl_2 solution and 5 mL of HCl to each flask. Dilute to volume and mix. The calibration solutions are identified as S_0 through S_4 and contain (0, 2.5, 5.0, 7.5, and 10.0) mg/L Mn, respectively.

17.4 Sample Dissolution and Dilution:

17.4.1 Dissolve samples in accordance with 9.1 – 9.1.4.

17.4.2 Primary Dilution for Samples Containing Less Than 0.10 % Manganese—Transfer the dissolved sample to a 100-mL volumetric flask. Add 4 mL of SrCl_2 solution, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

17.4.3 Primary Dilution for Samples Containing More Than 0.10 % Manganese—Transfer the dissolved sample to a 500-mL volumetric flask, add 20 mL of HCl, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

17.4.4 Secondary Dilution for Samples Containing Between 0.1 % and 4.0 % Manganese—Transfer 50 mL of the primary dilution solution into a 100-mL volumetric flask and add 4 mL

of SrCl_2 solution and 3 mL of HCl. Cool, dilute to mark, and mix. The dilution factor is $F = 2$.

17.5 Calibration, Determination, and Calculation—Complete the calibration, determination, and calculation in accordance with 9.2 through Section 11.

17.6 Precision and Bias:^{5,6}

17.6.1 Precision—Eleven laboratories in six countries cooperated in testing this method and obtained statistical information summarized in Table 7.

17.6.2 Bias—No information on the accuracy of this method is known because accepted reference standards were not used in the ILS. The user of the method is encouraged to use accepted reference materials, if available, to determine the accuracy of this method as applied in a specific laboratory.

18. Determination of Silicon

18.1 Parameters:

18.1.1 Wavelength—251.6 nm.

18.1.2 Flame: Nitrous oxide—Acetylene.

18.2 Reagents:

18.2.1 Lithium Chloride Solution—Dissolve 25 g of LiCl in 150 mL of warm water. Cool, transfer to a 200-mL volumetric flask, dilute to volume, and mix.

18.2.2 Silicon Stock Calibration Solution (1.000 g/L)—Transfer 1.000 g of elemental silicon powder (purity 99.9 % min) to a 250-mL PTFE beaker and add 20 mL of HNO_3 and wash the walls of the beaker with water. Add HF drop by drop to initiate and sustain a reaction (approximately 10 mL HF is required). After most of the silicon is dissolved, add 10 mL more of HF, cover the beaker, and keep below 50 °C until dissolution is complete. Transfer into a 1-L plastic volumetric flask, cool, dilute to volume, and mix. Store in a high-density polyethylene bottle.

18.2.3 Silicon Calibration Solution (100 mg/L)—Transfer, using a plastic pipette, 50 mL of the silicon stock calibration solution into a 500-mL plastic volumetric flask and add 5 mL HF (1 + 9), 10 mL of HCl. Dilute to volume and mix. Store in a high-density polyethylene bottle.

18.3 Silicon Calibration Solutions—Using a plastic burette, transfer to each of six 100-mL plastic volumetric flasks (0, 10.0, 20.0, 30.0, 40.0, and 50.0) mL, respectively, of the silicon calibration solution. To each flask add 2 mL of HCl and 5 mL HF (1 + 9) and dilute to about 80 mL. Add 3 mL of the LiCl solution to each volumetric flask. Dilute to volume and mix. The calibration solutions are identified as S_0 through S_5 and contain (0, 10.0, 20.0, 30.0, 40.0, and 50.0) mg/L Si, respectively.

TABLE 7 Results of Statistical Analysis—Manganese

Test Material ^A	Mean, %	Repeatability Index r (Practice E1601)	Reproducibility Index R (Practice E1601)
7013	0.058	0.0019	0.0048
3920	0.28	0.011	0.028
902	0.37	0.009	0.050
3927	0.39	0.014	0.048
825	0.74	0.043	0.088
7049	0.81	0.013	0.025

^A Nominal material compositions are summarized in Table 1.

18.4 *Sample Dissolution and Dilution:*

18.4.1 Transfer a 1.0-g sample weighed to the nearest milligram to a PTFE beaker. Add 15 mL of HCl and 5 mL of HNO₃. Apply sufficient heat to initiate and maintain the reaction until dissolution is complete. If the alloy resists dissolution, add HCl in 1-mL increments and continue heating to dissolve the sample. Cool the solution and wash the cover and the beaker walls with a minimum of water. Add 5 mL of HF (1 + 9) and allow to stand for 1 h, swirling intermittently.

18.4.2 *Primary Dilution for Samples Containing Less Than 0.50 % Silicon*—Transfer the dissolved sample to a 100-mL plastic volumetric flask. Add 2 mL of HCl and dilute to about 80 mL. Add 3 mL of LiCl solution, dilute to volume, and mix.

18.4.3 *Primary Dilution for Samples Containing More Than 0.50 % Silicon*—Transfer the dissolved sample to a 100-mL plastic volumetric flask, dilute to volume, and mix.

18.4.4 *Secondary Dilution for Samples Containing More Than 0.50 % Silicon*—Transfer 50 mL of the primary dilution solution into a 100-mL plastic volumetric flask and add 2 mL of HCl and 2.5 mL HF (1 + 9). Dilute to about 80 mL and mix. Add 3 mL of the LiCl solution, dilute to mark, and mix.

18.5 *Calibration, Determination, and Calculation*—Complete the calibration, determination, and calculation, in accordance with 9.2 through Section 11.

NOTE 8—To eliminate silica memory effects, the burner system must be preconditioned before analysis by aspirating HF (1 + 9). With the flame burning, aspirate this dilute acid solution until the original base line signal is restored, that is, when the silica deposit on the burner top has been volatilized. Then proceed with the aspiration of water as directed.

18.6 *Precision and Bias:*^{5, 6}

18.6.1 *Precision*—Six laboratories in four countries cooperated in testing this method and obtained statistical information summarized in Table 8.

18.6.2 *Bias*—No information on the accuracy of this method is known because accepted reference standards were not used in the ILS. The user of the method is encouraged to use accepted reference materials, if available, to determine the accuracy of this method as applied in a specific laboratory.

19. **Determination of Vanadium**

19.1 *Parameters:*

19.1.1 *Wavelength:* 318.4 nm.

19.1.2 *Flame:* Nitrous oxide—Acetylene.

19.2 *Reagents:*

19.2.1 *Strontium Chloride Ionization Buffer Solution*—Dissolve 113.5 g of SrCl₂ · 6H₂O in 400 mL hot water of (50 °C to 60 °C). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

TABLE 8 Results of Statistical Analysis—Silicon

Test Material ^A	Mean, %	Repeatability Index r (Practice E1601)	Reproducibility Index R (Practice E1601)
7049	0.336	0.019	0.045
902	0.344	0.011	0.043
825	0.403	0.019	0.069
3920	0.614	0.047	0.062
7013	0.721	0.049	0.078
3927	0.816	0.055	0.127

^A Nominal material compositions are summarized in Table 1.

TABLE 9 Results of Statistical Analysis—Vanadium

Test Material ^A	Mean, %	Repeatability Index r (Practice E1601)	Reproducibility Index R (Practice E1601)
925	0.042	0.017	0.102
NPK31	0.286	0.025	0.076
IN100	0.965	0.003	0.017

^A Nominal material compositions are summarized in Table 1.

19.2.2 *Vanadium Stock Calibration Solution* (1.000 g/L):

19.2.2.1 *Prepared from Vanadium Metal*—Dissolve 1.000 g of vanadium metal (99.9 % min) in 60 mL of HCl and 20 mL of HNO₃ and heat to complete the dissolution. Cool and transfer to a 1-L volumetric flask. Dilute to volume and mix. Store in a high-density polyethylene bottle.

19.2.2.2 *Prepared from Ammonium Metavanadate*—Dissolve 2.296 g of ammonium metavanadate (NH₄VO₃) in 400 mL of warm water. Transfer the warm solution to a 1-L volumetric flask and dilute with 400 mL of cold water. Add 50 mL of HCl and 10 mL of HNO₃. Cool, dilute to volume, and mix. Store in a high-density polyethylene bottle.

19.2.3 *Vanadium Calibration Solution* (250 mg/L)—Transfer 50 mL of the vanadium stock calibration solution into a 200-mL volumetric flask. Dilute to volume and mix. Store in a high-density polyethylene bottle.

19.3 *Vanadium Calibration Solution*—Transfer to each of five 100-mL volumetric flasks (0, 4.0, 8.0, 12.0, and 16.0) mL, respectively, of the vanadium calibration solution. To each flask add 3 mL of HCl, 1 mL of HNO₃, and 5 mL of the SrCl₂ solution to each volumetric flask. Dilute to volume and mix. The calibration solutions are identified as S₀ through S₄ and contain (0, 10.0, 20.0, 30.0, and 40.0) mg/L V, respectively.

19.4 *Sample Dissolution and Dilution:*

19.4.1 Dissolve samples in accordance with 9.1 – 9.1.3.

19.4.2 Cool to about 50 °C. Add 3 mL of HCl, 1 mL of HNO₃, and 20 mL of water and heat to dissolve salts.

19.4.3 *Primary Dilution for Samples Containing Less Than 0.35 % Vanadium*—Transfer the dissolved sample to a 100-mL volumetric flask. Add 5 mL of SrCl₂ solution, cool, dilute to volume, and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging. The dilution factor F = 1.

19.4.4 *Dilution for Samples Containing More Than 0.35 % Vanadium*—Transfer 20 mL of the primary dilution solution into a 100-mL volumetric flask, and add 4 mL of SrCl₂ solution, 3 mL of HCl, and 1 mL of HNO₃. Dilute to volume and mix. The dilution factor F = 5.

19.5 *Calibration, Determination, and Calculation*—Complete the calibration, determination, and calculation in accordance with 9.2 through Section 11.

19.6 *Precision and Bias:*^{5, 6}

19.6.1 *Precision*—Nine laboratories in five countries cooperated in testing this method and obtained statistical information summarized in Table 9.

19.6.2 *Bias*—No information on the accuracy of this method is known because accepted reference standards were not used in the ILS. The user of the method is encouraged to use

accepted reference materials, if available, to determine the accuracy of this method as applied in a specific laboratory.

20. Keywords

20.1 aluminum content; analysis; atomic absorption spectrometry; cobalt content; copper content; chromium content;

FAAS; flame atomic absorption spectrometry; iron content; manganese content; nickel; nickel alloys; silicon content; vanadium content

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