Designation: E 1835 – 96

Standard Test Method for Analysis of Nickel Alloys by Flame Atomic Absorption Spectrometry[1]

This standard is issued under the fixed designation E 1835; 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 and nickel-base alloys by flame atomic absorption spectrometric analysis for the following elements:

Element	Concentration Range, %	Section
Aluminum	0.2 to 4.0	12
Chromium	0.01 to 4.0	13
Cobalt	0.01 to 4.0	14
Copper	0.01 to 4.0	15
Iron	0.1 to 4.0	16
Manganese	0.1 to 4.0	17
Silicon	0.2 to 1.0	18
Vanadium	0.05 to 1.0	19

1.2 The concentration range of these elements can be expanded by the use of appropriate standards.

1.3 *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 E 50 and the warning statements included in this test method.

2. Referenced Documents

2.1 *ASTM Standards:*

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[2]

E 863 Practice for Describing Flame Atomic Absorption Spectroscopy Equipment[3]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[3]

E 1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analysis[3]

E 1812 Practice for Optimization of Flame Atomic Absorption Spectrophotometric Equipment[3]

2.2 *ISO Standards:*

ISO Guide 5725—Accuracy, Trueness, and Precision of Measurements, Methods and Results[4]

ISO 7530 Parts 2 through 9—Flame Atomic Absorption Spectrometric Analysis[4]

3. Summary of Test Method

3.1 The sample is dissolved in a mixture of hydrochloric acid and nitric acid. The solution is aspirated into an appropriate flame of an atomic absorption spectrometer. Measurement of the absorbance of the resonant line energy from the spectrum of the analyte is compared with that of calibration solutions.

4. Significance and Use

4.1 This test method is used for the analysis of nickel and nickel-base alloy samples by flame atomic absorption spectrometry to check compliance with compositional specifications. It is assumed that all who use the procedure will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that the work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices must be followed such as those described in Guide E 882.

4.2 *Interlaboratory Studies (ILS)*[5]—This test method was evaluated by a subcommittee within ISO Technical Committee 155 (ISO/TC 155/SC 4) on analysis of nickel alloys, in accordance with ISO Standard 5725. It was published as ISO Standard 7530, Parts 2 through 9. The ILS test data was not available for recalculation. The published ISO statistics are summarized separately for each analyte.

5. Apparatus

5.1 *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.

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

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[1] This test method is under the jurisdiction of ASTM Committee E-1 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] *Annual Book of ASTM Standards*, Vol 03.05.

[3] *Annual Book of ASTM Standards*, Vol 03.06.

[4] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[5] Supporting data are available from ASTM Headquarters. Request RR:E01-1018.

TABLE 1 Nominal Composition 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	...

5.3 For a general discussion of the instrumental requirements of flame atomic absorption see Practice E 863.

5.4 For a general discussion of apparatus requirements see Practices E 50.

6. Reagents

6.1 *Purity and Concentration of Reagents*—The purity and concentration of common chemical reagents shall conform to Practices E 50. The reagents should be free of or contain minimal amounts (<0.1µ g/g) of the analyte of interest.

6.2 *Calibration Solutions*—Made up for the individual analytes as described in Sections 12-19. They should be prepared to meet the guidelines of Practice E 1452.

6.3 *Matrix Modifiers and Ionization Buffers*—Made up for the individual analytes, where required, as described in Sections 12-19.

7. Sampling and Sample Preparation

7.1 Sampling and sample preparation is to be performed by procedures agreed upon between the buyer and the seller.

7.2 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.

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

7.4 The laboratory sample shall be cleaned by washing in pure acetone and then air dried.

7.5 If brazed alloy tools are used in the preparation of the sample, the sample shall be further cleaned by pickling in dilute nitric acid for a few minutes. The sample shall then be washed several times with water followed by several washes with acetone and air dried.

8. General Procedure

8.1 *Sample Dissolution*:

8.1.1 Transfer a 1.0-g sample, weighed to the nearest 1 mg, to a 600-mL beaker. Add 15 mL HCl and 5 mL HNO_3. Apply sufficient heat to initiate and maintain the reaction until the dissolution is complete. If the sample contains over 0.5 % silicone, a few drops of HF will considerably speed up the dissolution.

NOTE 2—**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.

8.1.2 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 3—For some alloys a 30-mL HCl—2-mL HNO_3 mixture is more effective. Pure nickel or nickel-copper alloys dissolve best in HNO_3 (1+1).

NOTE 4—The general method of dissolution can be modified as specified in the appropriate sections.

NOTE 5—If sample inhomogeneity is suspected, a larger mass of sample (10 to 50 g) may be taken for analysis. In that case, however, an aliquot portion corresponding to 1-g sample shall be taken from the solution and processed in accordance with the procedure given.

8.1.3 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.

8.1.4 Cool to about 50°C, add 5 mL hydrochloric acid and 20 mL water and heat to dissolve the salt.

8.1.5 Proceed as directed in Sections 12-19.

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

8.3 *Calibration Solutions*—Proceed as directed in Sections 12-19.

8.4 *Atomic Absorption Measurements*:

8.4.1 The wavelength of the spectral lines and the flame type to be used are listed in Sections 12-19.

8.4.2 Set the required instrument parameters in accordance with the manufacturer's recommendations and Practice E 1812. 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.

8.4.3 Ensure that the instrument meets the performance requirements given in Practice E 1812. Optimum settings for the operating parameters vary from instrument to instrument. Scale expansion may have to be used to obtain the required readability.

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

8.4.5 Aspirate water and zero the instrument.

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

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

8.4.8 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.

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

9. Preparation of Calibration Graphs

9.1 Plot the average instrument reading against the concentration of the analyte in the calibration solutions for each of the measurements.

NOTE 6—Some instruments may be adjusted to give a readout in concentration of the analyte. A graph of instrument response versus concentration should be plotted to check the validity of the readings.

9.2 Carry out measurements at least in triplicate.

10. Calculation

10.1 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.

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

$$\text{Analyte, \%} = (c\ V\ F)/10\ 000\ m \tag{1}$$

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. Report

11.1 Report at least the following information:

11.1.1 Designation of the test method used,

11.1.2 Results of the analysis,

11.1.3 Number of independent replications,

11.1.4 Any unusual features noted during the analysis, and

11.1.5 Any operation not included in this test method or described as optional.

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* (1.000 g/L)—Dissolve 1.000 g of aluminum (purity 99.9 % min) in 30 mL (1:1) of HCl in the presence of 1 drop of mercury. Filter the solution through a rapid filter paper. Wash the filter with 100 mL of warm water. Add 85 mL of HCl to the filtrate, cool and transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

NOTE 7—**Warning:** Mercury which acts a catalyst to help the dissolution of high-purity aluminum is highly poisonous and has an appreciable vapor pressure. It must be stored in strong, tightly closed containers. Liquid mercury must be transferred in such a manner that a spill can be contained and thoroughly cleaned up at once. Dispose of mercury in accordance with applicable regulations.

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, 15, 20, and 25 mL, respectively, of the aluminum calibration solution (12.2.3). Add 4 mL of the potassium chloride solution (12.2.1) and 4 mL of HNO_3 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 S_0 through S_5 and contain 0, 5.0, 10.0, 15.0, and 20.0 mg/L aluminum, respectively.

NOTE 8—It is important that all calibration solutions contain the same concentration (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 to a 400-mL PTFE beaker and add 15 mL of HCl and 5 mL of HNO_3. 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 potassium chloride solution (12.2.1). 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 HNO_3, and 4 mL of potassium chloride solution (12.2.1). 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 HNO_3, and 4 mL of potassium chloride solution (12.2.1). 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 HNO_3, and 4 mL of potassium chloride solution (12.2.1). 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) H_2SO_4 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.

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 HNO_3. Cool and dilute to volume and mix.

NOTE 9—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]

12.6.1 *Precision*—Six laboratories 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 employ 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 ($SrCl_2 \cdot 6H_2O$) in 400 mL of hot water (50 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 (1:1) of HCl. 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, 15, and 20 mL, respectively, of the chromium calibration solution (13.2.3). Add 4 mL of the strontium chloride solution (13.2.1) and 5 mL of HCl to each volumetric flask. Dilute to volume and mix. The calibration solutions are identified as S_0 through S_5 and contain 0, 2.5, 5.0, 7.5, 10, and 12.5 mg/L of chromium, respectively.

13.4 *Sample Dissolution and Dilution*:

13.4.1 Dissolve samples in accordance with 8.1-8.1.4.

13.4.2 *Primary Dilution for Samples Containing Less Than 0.10 % Chromium*—Transfer the dissolved sample (8.1.4) to a 100-mL volumetric flask. Add 4 mL of strontium chloride solution (13.2.1), 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 (8.1.4) 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 strontium chloride solution (13.2.1) 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 strontium chloride solution (13.2.1) 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 Paragraph 8.2 through Section 11.

13.6 *Precision and Bias:*[5]

13.6.1 *Precision*—Ten laboratories 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 employ accepted reference materials, if available, to determine the accuracy of this method as applied in a specific laboratory.

TABLE 2 Results of Statistical Analysis—Aluminum

Test Material[A]	Mean, %	Repeatability SD (S_r, ISO 5725)[B]	Reproducibility SD (S_R, ISO 5725)[C]	Reproducibility (R, ISO 5725)[D]	R_{rel5}
3927	0.109	0.003_2	0.004_4	0.015_9	14.6
3920	0.146	0.001_6	0.003_9	0.012_1	8.29
825	0.169	0.005_2	0.011_4	0.035_4	20.9
802	0.434	0.004_5	0.007_5	0.024_8	5.71
7049	0.972	0.008_7	0.008_6	0.034_5	3.55
7013	1.51	0.012_7	0.004_6	0.036_5	2.41

[A] Material compositions are summarized in Table 1.
[B] Equivalent to minimum standard deviation, S_M (see Practice E 1601).
[C] Equivalent to reproducibility standard deviation, S_R (see Practice E 1601).
[D] Equivalent to reproducibility index, R (see Practice E 1601).

TABLE 3 Results of Statistical Analysis—Chromium

Test Material[A]	Mean, %	Repeatability SD (S_r, ISO 5725)[B]	Reproducibility SD (S_R, ISO 5725)[C]	Reproducibility (R, ISO 5725)[D]	$R_{rel\%}$
902	5.16	0.034	0.102	0.30	5.81

[A] Material compositions are summarized in Table 1.
[B] Equivalent to minimum standard deviation, S_M (see Practice E 1601).
[C] Equivalent to reproducibility standard deviation, S_R (see Practice E 1601).
[D] Equivalent to reproducibility index, R (see Practice E 1601).

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 strontium chloride hexahydrate ($SrCl_2 \cdot 6H_2O$) in 400 mL of hot water (50 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 (1:1) of HCl. 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, 15, and 20.0 mL, respectively, of the cobalt calibration solution (14.2.3). Add 4 mL of the strontium chloride solution (14.2.1) and 5 mL of HCl to each volumetric 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 cobalt, respectively.

14.4 *Sample Dissolution and Dilution*:

14.4.1 Dissolve samples in accordance with 8.1-8.1.4.

14.4.2 *Primary Dilution for Samples Containing Less Than 0.10 % Cobalt*—Transfer the dissolved sample (8.1.4) to a 100-mL volumetric flask. Add 4 mL of strontium chloride solution (14.2.1), 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 (8.1.4) 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 strontium chloride solution (14.2.1) 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 strontium chloride solution (14.2.1) 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 8.2 through Section 11.

14.6 *Precision and Bias:*[5]

14.6.1 *Precision*—Twelve laboratories 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 employ 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 strontium chloride hexahydrate ($SrCl_2 \cdot 6H_2O$) in 400 mL of hot water (50 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 (1:1) of HNO_3. 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. Dissolve salts in 50 mL (1:1) of HCl, 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

TABLE 4 Results of Statistical Analysis—Cobalt

Test Material[A]	Mean, %	Repeatability SD (S_r, ISO 5725)[B]	Reproducibility SD (S_R, ISO 5725)[C]	Reproducibility (R, ISO 5725)[D]	$R_{rel\%}$
7049	0.007_2	0.000_2	0.000_9	0.002_6	36.1
902	0.046	0.001_2	0.001_7	0.006_0	13.0
825	0.067	0.000_6	0.002_1	0.006_0	8.95
3927	1.03	0.016	0.022	0.077	7.47
3920	2.01	0.029	0.0	0.082	4.08

[A] Material compositions are summarized in Table 1.
[B] Equivalent to minimum standard deviation, S_M (see Practice E 1601).
[C] Equivalent to reproducibility standard deviation, S_R (see Practice E 1601).
[D] Equivalent to reproducibility index, R (see Practice E 1601).

50 mL of the copper stock calibration solution (15.2.2) into a 1-L volumetric flask and add 50 mL of HCl. Dilute to volume and mix. Store in a high-density polyethylene bottle.

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 (15.2.3). Add 4 mL of the strontium chloride solution (15.2.1) and 5 mL of HCl to each volumetric 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 copper, respectively.

15.4 *Sample Dissolution and Dilution*:

15.4.1 Dissolve samples in accordance with 8.1-8.1.4.

15.4.2 *Primary Dilution for Samples Containing Less Than 0.10 % Copper*—Transfer the dissolved sample (8.1.4) to a 100-mL volumetric flask. Add 4 mL of strontium chloride solution (14.2.1), 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 (8.1.4) 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 (15.4.3) into a 100-mL volumetric flask, and add 4 mL of strontium chloride solution (15.2.1) 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 (15.4.3) into a 100-mL volumetric flask and add 4 mL of strontium chloride solution (15.2.1) 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 Paragraph 8.2 through Section 11.

15.6 *Precision and Bias*:[5]

15.6.1 *Precision*—Twelve laboratories 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 employ 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 strontium chloride hexahydrate ($SrCl_2 \cdot 6H_2O$) in 400 mL of hot water (50 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 (1:1) of HCl. 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 an 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 (16.2.2) into a 1-L volumetric flask and add 50 mL of HCl. Dilute to volume and mix. Store in a high-density polyethylene bottle.

16.3 *Iron Calibration Solutions*—Transfer to each of five 100-mL volumetric flasks 0, 5.0, 10, 15, and 20 mL, respectively, of the iron calibration solution (16.2.3). Add 4 mL of the strontium chloride solution (16.2.1) and 5 mL of HCl to each volumetric 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 of iron, respectively.

16.4 *Sample Dissolution and Dilution*:

16.4.1 Dissolve samples in accordance with 8.1-8.1.4.

16.4.2 *Primary Dilution for Samples Containing Less Than 0.10 % Iron*—Transfer the dissolved sample (8.1.4) to a 100-mL volumetric flask. Add 4 mL of strontium chloride solution (16.2.1), 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 (8.1.4) 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 (16.4.3) into a 100-mL volumetric flask and add 4 mL of strontium chloride solution (16.2.1) and 3 mL of HCl. Cool, dilute to mark, and mix. The dilution factor F = 2.

TABLE 5 Results of Statistical Analysis—Copper

Test Material[A]	Mean, %	Repeatability SD (S_r, ISO 5725)[B]	Reproducibility SD (S_R, ISO 5725)[C]	Reproducibility (R, ISO 5725)[D]	$R_{rel\%}$
902	0.036	0.000_4	0.001_2	0.003_5	9.72
3927	0.054	0.000_9	0.002_2	0.002_6	4.81
3920	0.11	0.002_9	0.005_0	0.016	14.5
7049	0.16	0.002_9	0.005_4	0.017	10.6
7013	0.20	0.002_1	0.004_2	0.013	6.5
825	162	0.005_7	0.018	0.053	3.27

[A] Material compositions are summarized in Table 1.
[B] Equivalent to minimum standard deviation, S_M (see Practice E 1601).
[C] Equivalent to reproducibility standard deviation, S_R (see Practice E 1601).
[D] Equivalent to reproducibility index, R (see Practice E 1601).

16.4.5 *Secondary Dilution for Samples Containing Between 0.8 and 4.0 % Iron*—Transfer 10 mL of the primary dilution solution (16.4.3) into a 100-mL volumetric flask and add 4 mL of strontium chloride solution (16.2.1) 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 Paragraph 8.2 through Section 11.

16.6 *Precision and Bias:*[5]

16.6.1 *Precision*—Eleven laboratories 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 employ 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 strontium chloride hexahydrate ($SrCl_2 \cdot 6H_2O$) in 400 mL of hot water (50 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 (1:1) of HCl 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 (17.2.2) into a 1-L volumetric flask and add 50 mL of HCl. Dilute to volume and mix. Store in a high-density polyethylene bottle.

17.3 *Manganese Calibration Solutions*—Transfer to each of five 100-mL volumetric flasks 0, 5.0, 10, 15, and 20 mL, respectively, of the manganese calibration solution (17.2.3). Add 4 mL of the strontium chloride solution (17.2.1) and 5 mL of HCl to each volumetric 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 manganese, respectively.

17.4 *Sample Dissolution and Dilution*:

17.4.1 Dissolve samples in accordance with 8.1-8.1.4.

17.4.2 *Primary Dilution for Samples Containing Less Than 0.10 % Manganese*—Transfer the dissolved sample (8.1.4) to a 100-mL volumetric flask. Add 4 mL of strontium chloride solution (17.2.1), 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 (8.1.4) 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 (17.4.3) into a 100-mL volumetric flask and add 4 mL of strontium chloride solution (17.2.1) 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 Paragraph 8.2 through Section 11.

17.6 *Precision and Bias:*[5]

17.6.1 *Precision*—Eleven laboratories 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 employ 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 lithium chloride 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 (18.2.2) into a 500-mL plastic volumetric flask and

TABLE 6 Results of Statistical Analysis—Iron

Test Material[A]	Mean, %	Repeatability SD (S_r, ISO 5725)[B]	Reproducibility SD (S_R, ISO 5725)[C]	Reproducibility (R, ISO 5725)[D]	$R_{rel\%}$
7013	0.21	0.002_3	0.006_3	0.019	9.04
3920	3.00	0.029	0.048	0.16	5.33

[A] Material compositions are summarized in Table 1.

[B] Equivalent to minimum standard deviation, S_M (see Practice E 1601).

[C] Equivalent to reproducibility standard deviation, S_R (see Practice E 1601).

[D] Equivalent to reproducibility index, R (see Practice E 1601).

TABLE 7 Results of Statistical Analysis—Manganese

Test Material[A]	Mean, %	Repeatability SD (S_r, ISO 5725)[B]	Reproducibility SD (S_R, ISO 5725)[C]	Reproducibility (R, ISO 5725)[D]	$R_{rel\%}$
7013	0.058	0.000_7	0.001_6	0.0048	8.3
3920	0.28	0.003_8	0.009_3	0.028	10.0
902	0.37	0.003_2	0.017	0.050	13.5
3927	0.39	0.005_0	0.016	0.048	12.3
825	0.74	0.015	0.027	0.088	11.9
7049	0.81	0.004_8	0.007_4	0.025	3.1

[A] Material compositions are summarized in Table 1.
[B] Equivalent to minimum standard deviation, S_M (see Practice E 1601).
[C] Equivalent to reproducibility standard deviation, S_R (see Practice E 1601).
[D] Equivalent to reproducibility index, R (see Practice E 1601).

add 5 mL (1:9) of HF, 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, 30, 40, and 50.0 mL, respectively, of the silicon calibration solution (18.2.3). To each flask add 2 mL of HCl and 5 mL of (1:9) HF and dilute to about 80 mL. Add 3 mL of the lithium chloride solution (18.2.1) 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 5.0 mg/L silicon, respectively.

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 HNO_3. 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 (1:9) of HF 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 (18.4.1) to a 100-mL plastic volumetric flask. Add 2 mL of HCl and dilute to about 80 mL. Add 3 mL of lithium chloride solution (17.2.1), and dilute to volume and mix.

18.4.3 *Primary Dilution for Samples Containing More Than 0.50 % Silicon*—Transfer the dissolved sample (18.4.1) 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 (18.4.3) into a 100-mL plastic volumetric flask and add 2 mL of HCl and 2.5 mL (1:9) of HF. Dilute to about 80 mL and mix. Add 3 mL of the lithium chloride solution (18.2.1), dilute to mark, and mix.

18.5 *Calibration, Determination, and Calculation*—Complete the calibration, determination, and calculation, in accordance with Paragraph 8.2 through Section 11.

NOTE 10—To eliminate silica memory effects, the burner system must be preconditioned before analysis by aspirating a dilute (1:9) solution of HF. 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]

18.6.1 *Precision*—Six laboratories 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 employ 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 strontium chloride hexahydrate ($SrCl_2 \cdot 6H_2O$) in 400 mL hot water of (50 to 60°C). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

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 HNO_3 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.

TABLE 8 Results of Statistical Analysis—Silicon

Test Material[A]	Mean, %	Repeatability SD (S_r, ISO 5725)[B]	Reproducibility SD (S_R, ISO 5725)[C]	Reproducibility (R, ISO 5725)[D]	$R_{rel\%}$
7049	0.336	0.006_6	0.014_4	0.044_3	13.2
902	0.344	0.003_9	0.014_9	0.043_5	12.6
825	0.403	0.006_6	0.023_6	0.069_4	17.2
3920	0.614	0.016_6	0.017_5	0.068_5	11.1
7013	0.721	0.017_4	0.021_3	0.077_3	10.7
3927	0.816	0.019_5	0.040_3	0.127	15.6

[A] Material compositions are summarized in Table 1.
[B] Equivalent to minimum standard deviation, S_M (see Practice E 1601).
[C] Equivalent to reproducibility standard deviation, S_R (see Practice E 1601).
[D] Equivalent to reproducibility index, R (see Practice E 1601).

19.2.2.2 *Prepared from Ammonium Metavanadate*—Dissolve 2.296 g of ammonium metavanadate (NH_4VO_3) 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 HNO_3. 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 (19.2.2.1 or 19.2.2.2) 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 mL, respectively, of the vanadium calibration solution (19.2.3). To each flask add 3 mL of HCl, 1 mL of HNO_3, and 5 mL of the strontium chloride solution (19.2.1) to each volumetric flask. Dilute to volume and mix. The calibration solutions are identified as S_0 through S_4 and contain 0, 10.0, 20.0, 30.0, and 40.0 mg/L vanadium, respectively.

19.4 *Sample Dissolution and Dilution*:

19.4.1 Dissolve samples in accordance with 8.1-8.1.3.

19.4.2 Cool to about 50°C. Add 3 mL of HCl, 1 mL of HNO_3, and 20 mL of water and heat to dissolve salts.

19.4.3 *Dilution for Samples Containing Less Than 0.35 % Vanadium*—Transfer the dissolved sample (19.4.2) to a 100-mL volumetric flask. Add 5 mL of strontium chloride solution (19.2.1), 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 *Primary Dilution for Samples Containing More Than 0.35 % Vanadium*—Transfer 20 mL of the primary dilution solution (19.4.2) into a 100-mL volumetric flask, and add 4 mL of strontium chloride solution, 3 mL of HCl, and 1 mL of HNO_3. 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 Paragraph 8.2 through Section 11.

19.6 *Precision and Bias:*[5]

19.6.1 *Precision*—Six laboratories 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 employ 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; flame atomic absorption spectrometry; iron content; manganese content; nickel; nickel alloys; silicon content; vanadium content

TABLE 9 Results of Statistical Analysis—Vanadium

Test Material[A]	Mean, %	Repeatability SD (S_r, ISO 5725)[B]	Reproducibility SD (S_R, ISO 5725)[C]	Reproducibility (R, ISO 5725)[D]	$R_{rel\%}$
925	0.042	0.001_2	0.005_9	0.003_4	8.09
NPK31	0.286	0.008_7	0.026_2	0.075_1	26.3
IN100	0.965	0.005_8	0.035_7	0.101_7	10.5

[A] Material compositions are summarized in Table 1.

[B] Equivalent to minimum standard deviation, S_M (see Practice E 1601).

[C] Equivalent to reproducibility standard deviation, S_R (see Practice E 1601).

[D] Equivalent to reproducibility index, R (see Practice E 1601).