



# Standard Test Method for Analysis of Nickel Alloys by Flame Atomic Absorption Spectrometry<sup>1</sup>

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 ( $\epsilon$ ) 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 spectrometric analysis (FAAS) for the following elements:

Element	Concentration 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 concentration range 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 E 50 and the warning statements included in this test method.

## 2. Referenced Documents

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

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

<sup>1</sup> 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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<sup>2</sup> 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.

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

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

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E 1812 Practice for Optimization of Flame Atomic Absorption Spectrometric Equipment<sup>3</sup>

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

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 E 135.

## 4. Summary of Test Method

4.1 The sample is dissolved in a mixture of HCl and HNO<sub>3</sub>. 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

<sup>3</sup> Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

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

**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	...

quality control practices must be followed such as those described in Guide E 882.

5.2 *Interlaboratory Studies (ILS)*<sup>5, 6</sup>—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 E 1601. 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 E 1601.

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

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

7.3 *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 HNO<sub>3</sub> 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 HNO<sub>3</sub>. 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 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 HNO<sub>3</sub> mixture is more effective. Nickel alloys dissolve best in HNO<sub>3</sub> (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 12-19.

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

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

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 12-19.

9.4 *Atomic Absorption Measurements*:

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

9.4.2 Set the required instrument parameters in accordance with the manufacturer's recommendations or 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.

9.4.3 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.

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

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 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:

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

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.

## 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 HCl (1 + 1) 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. (**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, 10.0, 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 HNO<sub>3</sub> 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, 20.0, and 25.0) mg/L Al, respectively.

NOTE 6—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, weighed to the nearest 1 mg, to a 400-mL PTFE beaker and add 15 mL of HCl and 5 mL of HNO<sub>3</sub>. 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 to approximately 60 mL. Cool and transfer to a 100-mL volumetric flask. Add 2.5 mL HCl, 4 mL HNO<sub>3</sub>, 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 to