



# 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 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:<sup>2</sup>

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

E 863 Practice for Describing Flame Atomic Absorption Spectroscopy Equipment

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

<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. Current edition approved Oct. 10, 1996. Published December 1996.

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

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

E 1812 Practice for Optimization of Flame Atomic Absorption Spectrometric Equipment

### 2.2 ISO Standards:

ISO Guide 5725—Accuracy, Trueness, and Precision of Measurements, Methods and Results<sup>3</sup>

ISO 7530 Parts 2 through 9—Flame Atomic Absorption Spectrometric Analysis<sup>3</sup>

## 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)*<sup>4</sup>—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

<sup>3</sup> Available from American National Standards Institute, 25W. 43rd St., 4th Floor, New York, NY 10036.

<sup>4</sup> Supporting data are available from ASTM International 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	...

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).

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 (<0.1 $\mu$  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<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 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<sub>3</sub> mixture is more effective. Pure nickel or nickel-copper alloys dissolve best in HNO<sub>3</sub> (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:

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

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. 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<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, 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<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 12.4.2 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 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