Designation: E2594 – 20

Standard Test Method for Analysis of Nickel Alloys by Inductively Coupled Plasma Atomic Emission Spectrometry (Performance-Based)[1]

This standard is issued under the fixed designation E2594; 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 describes the inductively coupled plasma atomic emission spectrometric analysis of nickel alloys, such as specified by Committee B02, and having chemical compositions within the following limits:

Element	Application Range (%)
Aluminum	0.01–1.00
Boron	0.001–0.050
Calcium	0.001–0.05
Carbon	0.10–0.20
Chromium	0.01–33.0
Cobalt	0.10–20.0
Copper	0.01–3.00
Iron	0.01–50.0
Lead	0.001–0.01
Magnesium	0.0001–0.100
Manganese	0.01–3.0
Molybdenum	0.01–30.0
Niobium	0.01–6.0
Nickel	25.0–80.0
Nitrogen	0.001–0.20
Oxygen	0.0001–0.003
Phosphorous	0.001–0.030
Sulfur	0.0001–0.010
Silicon	0.01–1.50
Tantalum	0.005–0.10
Tin	0.001–0.020
Titanium	0.001–6.0
Tungsten	0.01–5.0
Vanadium	0.01–1.0
Zirconium	0.01–0.10

1.2 The following elements may be determined using this test method. The test method user should carefully evaluate the precision and bias statements of this test method to determine applicability of the test method for the intended use.

Element	Quantification Range (%)
Aluminum	0.060–1.40
Boron	0.002–0.020
Calcium	0.001–0.003
Copper	0.010–0.52
Magnesium	0.001–0.10
Manganese	0.002–0.65
Niobium	0.020–5.5
Phosphorous	0.004–0.030
Tantalum	0.010–0.050
Tin	0.002–0.018
Titanium	0.020–3.1
Tungsten	0.007–0.11
Vanadium	0.010–0.50
Zirconium	0.002–0.10

1.3 This test method has only been interlaboratory tested for the elements and ranges specified. It may be possible to extend this test method to other elements or different quantification ranges provided that method validation is performed that includes evaluation of method sensitivity, precision, and bias as described in this document. Additionally, the validation study must evaluate the acceptability of sample preparation methodology using reference materials or spike recoveries, or both. The user is cautioned to carefully evaluate the validation data against the laboratory's data quality objectives. Method validation of scope extensions is also a requirement of ISO/IEC 17025.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in 8.2.6.3 and safety hazard statements are given in Section 9.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[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. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition

E88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis (Withdrawn 2019)[3]

E1479 Practice for Describing and Specifying Inductively Coupled Plasma Atomic Emission Spectrometers

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E2027 Practice for Conducting Proficiency Tests in the Chemical Analysis of Metals, Ores, and Related Materials

2.2 *ISO Standards:*[4]

ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories

ISO/IEC 17034 General Requirements for the competence of reference material producers

ISO Guide 31 Reference materials—Contents of certificates, labels and accompanying documentation

ISO Guide 98-3 Uncertainty of measurement—Part 3: Guide to the expression of uncertainty in measurement (GUM:1995), First Edition

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 Samples are dissolved in a mixture of mineral acids and the resulting solutions are measured using inductively coupled plasma atomic emission spectrometry.

5. Significance and Use

5.1 This test method for the chemical analysis of nickel alloys is primarily intended to test material for compliance with specifications such as those under jurisdiction of ASTM Committee B02. It may also be used to test compliance with other specifications that are compatible with the test method.

5.2 It is assumed that all who use this test method will be trained analysts capable of performing common laboratory procedures skillfully and safely, and that the work will be performed in a properly equipped laboratory.

5.3 This is a performance-based test method that relies more on the demonstrated quality of the test result than on strict adherence to specific procedural steps. It is expected that laboratories using this test method will prepare their own work instructions. These work instructions will include detailed operating instructions for the specific laboratory, the specific reference materials employed, and performance acceptance criteria. It is also expected that, when applicable, each laboratory will participate in proficiency test programs, such as described in Practice E2027, and that the results from the participating laboratory will be satisfactory.

6. Interferences

6.1 Practice E1479 describes the typical interferences encountered during the inductively coupled plasma spectrometric analysis of metal alloys. The user is responsible for ensuring the absence of or for compensating for interferences that may bias test results obtained using their particular spectrometer.

6.2 The use of an internal standard may compensate for the physical interferences resulting from differences between sample and calibration solutions transport efficiencies.

6.3 Shifts in background intensity levels because of, for example, recombination effects or molecular band contributions, or both, may be corrected by the use of an appropriate background correction technique. Direct spectral overlaps are best addressed by selecting alternative wavelengths. Spectral interference studies should be conducted on all new matrices to determine the interference correction factor(s) that must be applied to concentrations obtained from certain spectral line intensities to minimize biases. Some instrument manufacturers offer software options which mathematically correct for direct spectral overlaps, but the user is cautioned to carefully evaluate this approach to spectral correction.

6.4 Modern instruments have software that allows comparison of a sample spectrum to the spectrum obtained from a blank solution. The user of this test method must examine this information to ascertain the need for background correction and the correct placement of background points.

6.5 Table 1 suggests wavelengths that the user may use for analysis of nickel alloys. Each line was used by at least one laboratory during the interlaboratory phase of test method development and provided statistically valid results. Information for the suggested analytical wavelengths was collected from each laboratory and has been converted to wavelengths as annotated in the National Institute of Standards and Technology (NIST) Atomic Spectra Database.[5] In this database, wavelengths of less than 200 nm were measured in vacuum and wavelengths greater than or equal to 200 nm were measured in air. Software tables for individual instruments may list

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

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

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

[5] Ralchenko, Yu, Kramida, A. E., Reader, J., and NIST ASD Team (2008). *NIST Atomic Spectra Database* (version 3.1.5), National Institute of Standards and Technology, Gaithersburg, MD. Available online: http://physics.nist.gov/asd3 [2008, October 28].

TABLE 1 Suggested Wavelengths/Interferences

Element	Wavelength (nm)	Potential Interference
Aluminum	396.152	
Aluminum	394.401	Nickel
Aluminum	237.312	
Aluminum	176.638	
Aluminum	167.079	
Boron	182.641	Molybdenum, Cobalt, Chromium
Boron	182.591	Molybdenum, Cobalt, Chromium
Boron	136.246	Cobalt
Calcium	396.847	
Calcium	393.366	Cobalt
Copper	327.396	Titanium, Niobium, Gadolinium
Copper	224.700	Molybdenum, Iron
Copper	219.958	Tantalum
Copper	218.172	
Copper	217.894	
Copper	213.598	
Magnesium	383.829	
Magnesium	280.270	Cobalt
Magnesium	279.553	
Manganese	283.930	
Manganese	257.610	Cerium, Cobalt, Tungsten
Niobium	319.498	
Niobium	309.418	Chromium, Vanadium
Niobium	294.154	Vanadium
Niobium	269.706	
Niobium	210.942	
Phosphorous	178.766	
Phosphorous	178.284	Cobalt
Phosphorous	177.495	Nickel, Copper
Tantalum	263.558	Molybdenum
Tantalum	240.063	Cobalt, Chromium, Vanadium
Tantalum	226.230	
Tin	189.991	Titanium
Tin	175.800	
Tin	140.052	
Titanium	350.489	
Titanium	338.376	
Titanium	337.280	Niobium
Titanium	323.228	
Titanium	321.827	
Tungsten	207.912	
Tungsten	202.999	
Vanadium	437.924	
Vanadium	375.087	
Vanadium	309.311	
Vanadium	292.464	
Vanadium	292.402	
Zirconium	357.247	
Zirconium	343.823	Niobium
Zirconium	327.305	Chromium, Europium
Zirconium	256.887	

wavelengths somewhat differently, as instrument optical path atmospheric conditions may vary.

6.6 Information on potential spectral interfering elements was provided by the laboratories participating in the interlaboratory study and may have originated from sources such as recognized wavelength reference tables, instrument manufacturer's software wavelength tables, or an individual laboratory's wavelength research studies, or combinations thereof.

6.7 The user must verify that the selected wavelength performs acceptably in their laboratory, preferably during method validation (see Section 15). The user also may choose to use multiple wavelengths to help verify that line selection is optimized for the particular alloy being determined. It is recommended that when wavelengths and appropriate spectral corrections are determined, the user of this test method should specify this information or reference instrument programs that include this information in their laboratory analysis procedures.

7. Apparatus

7.1 *Inductively Coupled Plasma Atomic Emission Spectrometers*—Used to perform analysis by this test method may conform to the specifications given in Practice E1479. Suitability of a specific instrument for testing to this test method will be established using the performance criteria described in 12.1. The sample introduction system shall be capable of handling solutions containing up to 5 % HF.

7.2 *Sample Preparation Equipment*—Machine tools capable of removing surface oxides and other contamination from the as-received sample shall be used to produce chips or millings for analysis.

8. Reagents and Materials

8.1 *Reagents:*

8.1.1 *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.[6] 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.

8.1.2 *Purity of Water*—Unless otherwise indicated, references to water shall mean reagent water as defined by Type II of Specification D1193. The water purification method used must be capable of removal of all elements in concentrations that might bias the test results.

8.1.3 *Internal Standard*—The use of an internal standard is optional. However, the use of an internal standard may compensate for the physical interferences resulting from differences in sample and calibration solutions transport efficiency.

8.2 *Calibration Solutions:*

8.2.1 In this test method, calibration is based on laboratory-prepared, alloy matrix-matched calibration solutions. Alloy matrix-matched calibration solutions are solutions that contain the approximate amounts of the major alloying elements nickel, chromium, cobalt, molybdenum, and iron found in typical sample solutions. They are intended to model the physical behavior of sample solutions in the plasma. The matrix solutions are prepared with starting materials of known purity and are then spiked with aliquots of single element certified reference material (CRM) solutions that contain the analytes to be quantified. The CRMs shall be compliant with

[6] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC, www.chemistry.org. 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, http://www.usp.org.