Designation: E3047 – 22

Standard Test Method for Analysis of Nickel Alloys by Spark Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation E3047; 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 method describes the spark atomic emission spectrometric (Spark-AES) analysis of nickel alloys, such as those specified by Committee B02, having chemical compositions within the following limits:

Element	Application Range (Mass Fraction, %)
Aluminum	0.005-6.00
Boron	0.001-0.10
Carbon	0.005-0.15
Chromium	0.01-33.00
Copper	0.01-35.00
Cobalt	0.01-25.00
Iron	0.05-55.00
Magnesium	0.001-0.020
Manganese	0.01-1.00
Molybdenum	0.01-35.00
Niobium	0.01-6.0
Nickel	25.00-100.0
Phosphorous	0.001-0.025
Silicon	0.01-1.50
Sulfur	0.0001-0.01
Titanium	0.0001-6.0
Tantalum	0.01-0.15
Tin	0.001-0.020
Tungsten	0.01-5.0
Vanadium	0.0005-1.0
Zirconium	0.01-0.10

1.2 The following elements may be determined using this method.

Element	Quantification Range (Mass Fraction, %)
Aluminum	0.010-1.50
Boron	0.004-0.025
Carbon	0.014-0.15
Chromium	0.09-20.0
Cobalt	0.05-14.00
Copper	0.03-0.6
Iron	0.17-20
Magnesium	0.001-0.03
Manganese	0.04-0.6
Molybdenum	0.07-5.0
Niobium	0.02-5.5
Phosphorous	0.005-0.020
Silicon	0.07-0.6
Sulfur	0.002-0.005
Tantalum	0.025-0.15
Tin	0.001-0.02
Titanium	0.025-3.2
Tungsten	0.02-0.10
Vanadium	0.005-0.25
Zirconium	0.01-0.05

1.3 This method has been interlaboratory tested for the elements and quantification ranges specified in 1.2. The ranges in 1.2 indicate intervals within which results have been demonstrated to be quantitative. It may be possible to extend this method to other elements or different composition ranges provided that a method validation study as described in Guide E2857 is performed and that the results of this study show that the method extension is meeting laboratory data quality objectives. Supplemental data on other elements not included in the scope are found in the supplemental data tables of the Precision and Bias section.

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

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E29 Practice for Using Significant Digits in Test Data to

[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.

Determine Conformance with Specifications
E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E305 Practice for Establishing and Controlling Spark Atomic Emission Spectrochemical Analytical Curves
E406 Practice for Using Controlled Atmospheres in Atomic Emission Spectrometry
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1257 Guide for Evaluating Grinding Materials Used for Surface Preparation in Spectrochemical Analysis
E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis (Withdrawn 2019)[3]
E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
E2857 Guide for Validating Analytical Methods
E2972 Guide for Production, Testing, and Value Assignment of In-House Reference Materials for Metals, Ores, and Other Related Materials

2.2 *ISO Standards:*[4]

ISO/IEC Guide 98-3:2008 Uncertainty of Measurement—Part 3: Guide to the Expression of Uncertainty in Measurement (GUM:1995)

3. Terminology

3.1 *Definitions*—For definitions of terms used in this Practice, refer to Terminology E135.

4. Summary of Test Method

4.1 A controlled electrical discharge is produced in an argon atmosphere between the prepared flat surface of a specimen and the tip of a counter electrode. The energy of the discharge is sufficient to ablate material from the surface of the specimen, break the chemical or physical bonds, and cause the resulting atoms or ions to emit radiant energy. The radiant energy is dispersed by a grating and energies of selected analytical wavelengths and the internal standard wavelength(s) are converted into electrical signals by either photomultiplier tubes (PMTs) or a suitable solid-state detector. The detected analyte signals are integrated and converted to an intensity value. A ratio of the detected analyte intensity and the internal standard signal may be made. A calibration is made using a suite of reference materials with compositional similarity to the specimens being analyzed. Calibration curves plotting analyte intensity (intensity ratio) versus analyte mass fraction are developed. Specimens are measured for analyte intensity and results in mass fraction are determined using the calibration curves.

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 compositional specifications such as those under jurisdiction of 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 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 It is expected that laboratories using this method will prepare their own work instructions. These work instructions will include detailed operating instructions for the specific laboratory including information such as applicable analytical methods, drift correction (standardization) protocols, verifiers, and performance acceptance criteria.

6. Interferences

6.1 When possible, select analytical wavelengths which are free from spectral interferences. However, this is not always possible, and it may be necessary to apply interelement corrections to account mathematically for the effect of the interference on the measured intensities. If interference corrections are necessary, refer to Practice E305 for detailed information on the various techniques used to calculate interference corrections.

6.2 Table 1 lists analytical wavelengths routinely used for analysis of nickel alloys. For consistency of expression, the wavelengths are all listed as stated in the National Institute of Standards and Technology (NIST) Atomic Spectroscopy Database.[5] In the NIST wavelength table, wavelengths < 200 nm are as determined in a vacuum and wavelengths of ≥ 200 nm are as determined in air. Interference corrections, as reported by the interlaboratory study participants, are also indicated. It is not implied that analyses using this test method must be made with the same atmospheric conditions as stated for the NIST listed wavelengths. Performance of the analytical wavelength selected should be evaluated during method development for sensitivity and potential interferences.

7. Apparatus

7.1 *Spark Atomic Emission Spectrometer,* containing the following basic components.

7.1.1 *Spark Source*—The excitation source uses computer software which typically produces: *(1)* a high-energy pre-spark (of some preset duration), *(2)* a spark-type discharge (of some preset duration), *(3)* an arc type discharge (of some preset duration), and *(4)* a spark-type discharge, during which, time resolved measurements are made for improved detection limits, (this may be optional on some instruments). The counter-electrode serves as a conduction path for the high voltage discharge. The counter-electrode configuration/composition is typically specified by the instrument manufacturer.

7.1.2 *Analytical Stand*—Capable of supporting the specimen and counter-electrode in a manner such that the discharge

[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] Kramida, A., Ralchenko, Yu., Reader, J., and NIST ASD Team (2014). NIST Atomic Spectra Database (ver. 5.2), [Online]. Available: http://physics.nist.gov/asd [2015, July 29]. National Institute of Standards and Technology, Gaithersburg, MD.