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.

TABLE 1 Analytical Wavelengths for the Analysis of Nickel Alloys and Potential Interferences

Element	Wavelength, nm	Potential Interference	Element	Wavelength, nm	Potential Interference
Aluminum	308.22	Cr, Mo, Nb, Ti	Nickel	150.00	
Aluminum	309.28	Cu, Fe, Mo, Nb	Nickel	166.66	
Aluminum	394.40	Co, Cr, Cu, Fe, Mo, Nb, Si, W	Nickel	182.31	
Aluminum	616.43		Nickel	208.08	
Arsenic	189.04	Fe	Nickel	210.58	
Boron	182.64	Co, Cr, Fe, Mn, Mo, Ti, W	Nickel	214.78	
Boron	345.13		Nickel	218.55	
Calcium	396.85		Nickel	226.14	
Calcium	393.37	Fe	Nickel	232.27	
Carbon	193.09	Al, Fe	Nickel	243.79	
Carbon	165.70	Fe	Nickel	282.13	
Cobalt	228.62	Cr, Fe, Mo, Nb, W, Ti	Nickel	301.91	
Cobalt	258.03	Fe, Mo, Nb, W	Nickel	304.50	
Cobalt	345.35	Cr, Fe, Mo, Nb, Ti, W	Nickel	309.71	
Cobalt	384.55	Cr, Fe, Mo, Ti, W	Nickel	310.55	
Cobalt	184.59	Al, Fe, Ti	Nickel	346.95	
Chromium	267.72	Cu, Mo, Nb	Nickel	376.95	
Chromium	298.92	Al, Co, Fe, Ti, W	Nickel	380.71	
Copper	199.97	Fe, Mo, Nb	Nickel	471.44	
Copper	212.30	Co, Mn, Ti, Si, Sn	Phosphorous	177.49	Cu, Mo, Nb, W
Copper	224.26	Ni, W	Phosphorous	178.28	Cr, Fe, Mo, Nb, W
Copper	282.44		Silver	338.29	Co, Cr
Copper	324.75	Fe, Nb, W	Silver	328.07	Mo
Copper	510.55	Co, Cr, Mo, Nb, W	Silicon	212.41	Cr, Co, Fe, Mo, Nb, W
Iron	260.02	Co, Cr, Cu, W	Silicon	288.16	Al, Cr
Iron	273.07	Co, Cr, Ti, W, Mo, Nb	Sulfur	180.73	Al, Co, Cr, Mn, Mo, Nb, Ni, Ti, W
Iron	275.57	Al, Co, Cu, Mn, Mo, Nb Ti, W	Tantalum	240.06	Co
Iron	371.99		Tantalum	293.27	Cr, Nb, Ni, W
Iron	492.39		Tantalum	331.12	Cr, Nb, W, Zr
Magnesium	279.08	Fe	Tin	189.99	Cr, Mo, Nb, Ti, V
Manganese	263.82	Al, Cr, Fe, Mo, W	Tin	300.91	Cr, Fe, Mo
Manganese	273.09	Cr, Fe, Ti	Tin	317.50	Fe
Manganese	293.93		Titanium	308.81	Co, Cu, Fe, Mo, W
Molybdenum	202.03	Cr, Mn, Ni, W	Titanium	324.20	Co, Cr, Fe, Mo, Nb, W
Molybdenum	281.61	Al, Co, Cr, Fe	Vanadium	311.07	Al, Co, Cr, Cu, Fe, Mo, Nb, Ti,
Molybdenum	290.91	Cr, Fe, W	Tungsten	220.45	Al, Co, Cr, Mo
Molybdenum	308.76	Cr, Fe, W	Tungsten	400.90	Co, Cr, Fe, Mo, Nb, Ti
Molybdenum	369.26	Fe	Zirconium	343.82	Co, Cr, Fe, Mo, Ta, Ti, W
Niobium	319.50	W	Zirconium	349.62	Co, Cr, Mn, Mo
			Zirconium	468.84	

of the spark source may conduct through the flat, uniform surface of a prepared specimen. Additionally, the stand is designed to work in conjunction with the gas flow system.

7.1.3 *Gas Flow System*—Designed to deliver pure argon gas to the spark discharge, specimen interface region. Use the minimum argon purity specified by the instrument manufacturer. Refer to Practice E406 for practical guidance on the use of controlled atmospheres.

7.1.4 *Spectrometer*—Having acceptable dispersion, resolution, and wavelength coverage for the determination of nickel alloys. Table 1 provides guidance on the wavelengths that may be required.

7.1.5 *Optional Optical Path Purge or Vacuum System*—Designed to enhance ultraviolet wavelength sensitivity by either purging the optical path with a UV-transparent gas or by evacuating the optical path to remove air. The UV-transparent gas shall meet the manufacturer's minimum suggested purity requirements. Typically, the sum of the residual O_2 and H_2O impurities in the UV-transparent gas should not exceed 2 µmol/mol (ppm).

7.1.6 *Measuring and Control Systems*—Designed to convert emitted light intensities to a measurable electrical signal. These systems will consist of either a series of photomultiplier tubes (PMTs) or solid-state photosensitive arrays Charge Coupled Device (CCD) or Charge Injection Device (CID) and integrating electronics. Dedicated computer software is used to control analytical method conditions, source operation, data acquisition, and the conversion of intensity data to mass fraction.

7.1.7 *Other Software*—Designed to coordinate instrument function. At a minimum, the instrument's software should include functions for calibration, instrument drift correction

(standardization) and sample measurement. Additional software features may include functionality for tasks such as control charting.

7.2 *Specimen Preparation Equipment*—A grinder, milling machine or lathe capable of machining nickel alloy specimens to produce a clean, flat analytical surface.

8. Reagents and Materials

8.1 *Reference Materials (RMs):*

8.1.1 *Certified Reference Materials (CRMs)* should be used as calibration reference materials (RMs), if available. These CRMs should be of similar composition to the alloys being analyzed. If CRMs are not available for the element or alloy being analyzed or if available CRMs do not adequately cover the required analytical range, it is acceptable to use other reference materials for calibration.

8.1.2 *In-house RMs*—Some laboratories may have the resources to produce in-house RMs for nickel alloys. It is acceptable to use these RMs for calibration of Spark-AES instruments provided that the in-house RMs have been developed following technically sound development protocols, such as those described in Practice E2972.

8.1.3 *Instrument Manufacturer Provided RMs*—Some manufacturers perform factory calibrations which may include RMs owned by the manufacturer. The laboratory should make reasonable attempts to secure certificates of analysis for each of these RMs and to evaluate the acceptability of these certificates in conjunction with the laboratory's quality policies.

8.2 *Grinding Media*—If grinding is used as the specimen preparation technique, belts or disks of appropriate grit shall be provided. Aluminum oxide and silicon carbide based abrasive materials have been found to be acceptable for grinding nickel alloys. Typically, 60 grit or finer abrasive materials are found to be acceptable. Guide E1257 may be consulted for guidance to evaluate grinding materials.

8.3 *Lathe/Milling Tooling*—If lathe turning or milling is used for specimen preparation then tools appropriate for cutting nickel alloys shall be provided.

8.4 *Drift Correction (Standardization) Samples*—Select a suite of drift correction samples. This suite of samples should be of similar composition to the alloys being analyzed and should contain analyte levels near the lower and upper extremes of the calibration range for each analyte. Drift correction involves a calculated adjustment to calibration slope and intercept based on intensity changes observed for the measured drift correction samples. Although in some cases CRMs may be used for this purpose, it is not necessary or desired that CRMs be used, as drift correction does not involve calibration. Refer to Practices E305 and E1329 for a more detailed discussion of the use of drift correction (standardization) samples in Spark-AES analysis.

8.5 *Verifiers*—The verifiers should be of similar composition to the unknowns. Additionally, they should contain analytes in sufficient quantity to display a significant intensity response when ablated, so calibration drift may be quantified. Refer to Practices E305 and E1329 for a more detailed discussion of the use of verifiers in Spark-AES analysis.

9. Hazards

9.1 The excitation sources present a potential electrical shock hazard. The sample stand shall be provided with a safety interlock system to prevent energizing the source whenever contact with the electrode is possible. The instrument should be designed so access to the power supply is also restricted using safety interlocks.

9.2 Exhaust gas containing fine metallic dust generated by the excitation process may be a health hazard. Therefore, the instrument should be designed with an exhaust system to remove this dust in a safe manner. Some instruments are equipped with a filtration system designed for this purpose. An acceptable alternative to the filtration system would be a ventilation system that exhausts the powder to a "safe" area outside of the laboratory. If a filtration system is used, it should be maintained according to the manufacturer's recommendations.

9.3 If the filtration system includes filters, the filters used to collect the internal dust are likely exposed to an oxygen-depleted atmosphere. Sudden exposure of the filter to air may create a fire hazard. The laboratory should assess the risks associated with used filter disposal.

10. Sampling, Test Specimens, Test Specimen Preparation

10.1 Laboratories should follow written practices for sampling and preparation of test specimens.

10.2 Test specimens should be free of porosity or inclusions.

10.3 The test specimen must fit the specimen stand being used and must be large enough to cover the specimen orifice on the analytical stand of the instrument.

10.4 The test specimen configuration must be compatible with the selected specimen preparation equipment.

10.5 Prepare the specimen surface by either grinding, milling or lathe turning to produce a clean, flat analytical surface. A visual inspection for flatness is acceptable. Prepare the specimens, drift correction (standardization) samples, calibration RMs, and verifiers using the same procedure.

11. Preparation of Apparatus/Method Development

11.1 Analytical instrumentation and specimen preparation equipment shall be installed following the manufacturer recommendations.

11.2 Specify the following parameters into the instrument software.

11.2.1 The excitation source conditions.

11.2.2 The analytical wavelengths and measurement conditions to be used for measurement.

11.2.3 The internal standard wavelength(s) and associated measurement parameters, if intensity ratio is to be used as the expression for the measurement response. Nickel is typically used as the internal standard for the analysis of nickel alloys.

11.2.4 Drift correction (standardization) sample identification and associated measurement parameters. If possible, each analyte should be assigned a drift correction (standardization) sample containing analyte mass fractions near the anticipated calibration lower and upper extremes. If the software supports

the use of multiple point drift correction (standardization), specify additional drift correction (standardization) samples, as necessary.

11.2.5 Calibration RM identification, analyte mass fractions and associated measurement parameters.

11.2.6 Appropriate reporting parameters such as result format, unit of measure, reporting order, report destination, etc.

11.2.7 Optimize source operating conditions, analyte lines, and measuring conditions by performing test burns on calibration RMs in order to assess the sensitivity and precision of the selected measuring conditions.

11.2.8 An examination of intensity data from the test burns should suggest that the selected measurement conditions are acceptable. Examine the intensity data for these attributes.

11.2.8.1 There is a change in response for increasing analyte mass fraction.

11.2.8.2 The % relative standard deviation (RSD) of the intensity multiplied by the analyte mass fraction of a calibration RM in the analytical range yields an estimated analyte standard deviation that is consistent with the laboratory's measurement quality objectives.

11.2.8.3 Ultimately, the acceptability of the selected measurement method parameters will be demonstrated by the method validation study.

11.2.9 The laboratory should make a copy of the analytical parameters offline for backup in the event of instrument database corruption.

12. Calibration

12.1 Select calibration RMs which adequately define the instrument response across the range of expected analyte mass fractions. Practice E305 provides general guidance about selection of RMs for calibration. The quality and number of these calibration RMs will have a bearing on the quality of the calibration curves obtained. The interlaboratory study made during the development of this method demonstrated cases where laboratories clearly did not have robust calibrations covering the full range of specimen compositions which caused significant calibration biases and outlying data for some elements.

12.2 Prepare the drift correction (standardization) samples and calibration RMs per 10.5.

12.3 Measure the drift correction (standardization) samples. Measure each sample for a minimum of three excitation cycles (burns), re-positioning the sample between burns so that the ablated areas of the burns do not overlap. Burns should be made approximately 6 mm from the edge of the sample. If burns are to be made near the center of the sample, consider the metallurgical condition of the sample, since chill-cast samples may have a shrinkage cavity near the center of the casting. Observe the % RSD obtained for the burns. The scope elements listed in the method quantification range will typically exhibit < 3 % RSD for the average of the burns.

12.4 Prepare the calibration RMs and test specimens per 10.5.

12.5 Measure each calibration RM for a minimum of three burns, re-positioning the calibration RM between burns so that the ablated areas of the burns do not overlap. Burns should be made approximately 6 mm from the edge of the calibration RM. If burns are to be made near the center of the calibration RM, consider the metallurgical condition of the RM, since chill-cast RMs may have a shrinkage cavity near the center of the casting. Observe the % RSD calculated for the three burns. The scope elements listed in the method quantification range will typically exhibit < 3 % RSD for the average of the burns.

12.6 Calibration curves are calculated by calculating an expression of intensity (raw intensity or ratio of raw intensity to internal standard intensity) versus analyte mass fraction for the calibration RMs. Creation of the calibration curves will involve multivariate regression analysis, including correction for potential interferences. As necessary, apply interelement corrections to mathematically correct for interferences. Refer to Practice E305 for a detailed discussion on calculating calibration curves for Spark-AES.

13. Procedure

13.1 Place a prepared specimen over the orifice in the instrument analytical stand. There should be no gaps at the edge of the specimen. Choose the location for measurement to be approximately 6 mm from the edge of the specimen. If burns are to be made near the center of the specimen, consider the metallurgical condition of the specimen, since chill-cast specimens may have a shrinkage cavity near the center of the casting.

13.2 Perform a minimum of two separate burns on the specimen, re-positioning the specimen between burns so that the ablated areas of the burns do not overlap.

13.3 Examine the calculated % RSD for the average of the burns. The scope elements listed in the method quantification range will typically exhibit < 3 % RSD for the average of the burns. The laboratory may choose to make additional burns to get a better estimate of the average and its variance.

14. Verification, Drift Correction (Standardization), Type Standardization

14.1 The laboratory shall establish procedures for control of instrument response drift. These procedures should involve the use of a verifier and control chart to monitor drift. Refer to Practice E1329 for guidance in the preparation and use of control charts. Use control chart limits equal to two times or three times the standard deviation (2 s or 3 s) to indicate the need for drift correction (standardization).

14.2 If the instrument software allows, it is acceptable to apply the control strategy using the software. Calculate control limits for the verifier as described in Practice E1329 and record in the software.

14.3 Prepare control charts/control limits for each verifier/element combination.

14.4 The laboratory shall establish a frequency of analysis for the verifier. Once a verifier control strategy is established, analyze the verifier in accordance with the established protocol to evaluate instrument response drift.

14.5 Drift correct (standardize) the instrument when the verifier measurement indicates that the spectrometer has drifted to the point that one or more elements exceed the established 2 s or 3 s control limits. Update the drift correction (standardization) using the drift correction (standardization) samples established in 12.3.

14.6 Laboratories may wish to utilize type standardization samples to improve the accuracy of correcting calibration drift.

14.6.1 Reference materials used for type standardization updates must be compositionally very similar to the unknown samples. Take care to properly perform type standardization updates to prevent errant correction results.

14.6.2 Create the type standard as required by the software and analyze it a minimum of three excitations.

14.6.3 Evaluate the type standardardization by analyzing the verifier to ensure statistical control.

14.7 Users of this method are discouraged from using CRMs as drift correction, verifier or type standardization samples.

15. Method Validation

15.1 A laboratory using this method for the first time shall provide method validation data to demonstrate that the method as applied in their laboratory is yielding repeatable, unbiased results.

15.2 Guide E2857 should be consulted for guidance in performing the method validation study. It suggests multiple means of validating analytical methods. For this Spark-AES validation study, the minimum expectation is that the laboratory will prepare and analyze solid CRMs or RMs or both using the method to obtain the necessary validation data. Ideally these will be RMs that are independent of the calibration. The precision and bias data obtained for these RMs must then be compared to the precision and bias data stated in this method. The interlaboratory study associated with development of this test method clearly showed biases related to measurement of specimens with analyte composition near the extremes of available calibration RMs. The laboratory should verify calibration robustness by analyzing RMs near the extremes of the working range of the calibration.

15.3 If the validation exercise yields precision and bias data worse than given in the Precision and Bias section of this method, the laboratory should attempt to identify and correct any problems associated with their application of this method.

15.4 Ultimately, the method user must weigh customer requirements and the laboratory's data quality objectives to justify acceptance of the method validation data.

15.5 The method validation study shall be documented.

16. Calculations

16.1 Analyte results for the unknowns are determined by comparing the intensity (raw intensity or ratio of raw intensity to internal standard intensity) obtained for the specimen measurements to the calibration curve.

16.2 All calculations may be performed using the instrument software. Calculate the mean of the results of the individual measurements of each specimen and report the result as a mass fraction, either in % or mg/kg.

16.3 Rounding of test results obtained using this Test Method shall be performed in accordance with Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification.

17. Report

17.1 Results shall be reported following laboratory internal requirements.

17.2 When uncertainty estimates are required, results may be reported in accordance with the guidance provided in ISO/IEC Guide 98-3:2008. In this document, it is explained that the user must obtain an estimate of the overall uncertainty of the result and express that uncertainty as an expanded uncertainty $U = ku_c$, where u_c is a combined uncertainty expressed at the level of one standard deviation (1 s), and k is an expansion factor typically chosen as $k = 2$ to approximate a 95 % level of confidence. It is suggested that the laboratory include all significant sources of uncertainty in their estimate of the combined uncertainty. Express the value of U with 2 significant digits. Then, express the reported result to the same number of significant digits.

18. Precision and Bias

18.1 *Precision*—The precision of this test method is based on an interlaboratory study conducted in 2014. Ten laboratories participated in this study, testing thirteen total materials of five different alloys for specified elemental contents. One laboratory submitted two datasets, making eleven datasets available for statistical analysis in some cases. Not every laboratory was able to submit results for every alloy/element combination, however each "test result" reported represents an individual determination, and all participants were asked to report triplicate test results for each alloy/element pairing. The interlaboratory study was conducted in accordance with Practice E1601, the details of which are given in RR:E01-1124.[6] Statistical analysis of the data was performed in accordance with Practice E691. The precision statement was determined through statistical examination of usable test results, submitted by ten laboratories (up to eleven datasets), measuring twenty elements, in thirteen test materials.

18.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on an identical test material within short intervals of time would, in the normal and correct operation of the test method, exceed the following values only in one instance in 20.

18.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

18.1.1.2 Repeatability limits are listed in Tables 2-25.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1124. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Aluminum (mass fraction %)

Note—Data from up to N=11 datasets utilized.

Material	N	Average[A] $\bar{x}$	Certified or Reference Value	Bias %	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
WT48 (718 Alloy)	10	0.181			0.0013	0.038	0.0036	0.11
WN53 (718 Alloy)	11	0.456			0.0030	0.012	0.0085	0.034
NIST1249 (718 Alloy)	11	0.566	0.5682	-0.4%	0.0031	0.017	0.0086	0.047
NIST1244 (600 Alloy)	10	0.251	0.252	-0.6%	0.0022	0.016	0.0061	0.044
Brammer BS600-4 (600 Alloy)	10	0.0528	0.060	-11.9%	0.00047	0.0098	0.0013	0.028
WT71 (600 Alloy)	10	1.253			0.020	0.097	0.055	0.27
WASP79 (Waspaloy)	10	1.431			0.014	0.094	0.038	0.26
NIST1243 (Waspaloy)	10	1.241	1.23	0.9%	0.017	0.060	0.048	0.17
ARMI62B (Waspaloy)	10	1.354	1.38	-1.9%	0.013	0.063	0.037	0.18
NIST1230 (A286 Alloy)	10	0.244	0.249	-2.0%	0.0034	0.029	0.0095	0.080
ARMI26C (A286 Alloy)	10	0.121	0.12	0.5%	0.0014	0.015	0.0038	0.042
A286-48 (A286 Alloy)	10	0.469			0.0045	0.039	0.013	0.11
Brammer BS200-4	10	0.0076	0.0057	33.1%	0.00036	0.0018	0.0010	0.0049

[A]The average of the laboratories' calculated averages.

TABLE 3 Boron (mass fraction %)

Note—Data from up to N=11 datasets utilized.

Material	N	Average[A] $\bar{x}$	Certified or Reference Value	Bias %	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
WT48 (718 Alloy)	11	0.0171			0.0010	0.0040	0.0028	0.011
WN53 (718 Alloy)	9	0.00059			0.00004	0.00074	0.00010	0.0021
NIST1249 (718 Alloy)	10	0.00234	0.0023	1.6%	0.00003	0.00036	0.00007	0.0010
NIST1244 (600 Alloy)	9	0.00346	0.00283	22.2%	0.00010	0.00080	0.00028	0.0022
Brammer BS600-4 (600 Alloy)	9	0.0066	0.0060	10.5%	0.00012	0.0021	0.00035	0.0059
WT71 (600 Alloy)	8	0.00091			0.00003	0.00057	0.00008	0.0016
WASP79 (Waspaloy)	8	0.00225			0.00004	0.00043	0.00012	0.0012
NIST1243 (Waspaloy)	8	0.00512	0.00494	3.7%	0.00009	0.00020	0.00024	0.00056
ARMI62B (Waspaloy)	8	0.00488	0.005	-2.3%	0.00016	0.00025	0.00046	0.00071
NIST1230 (A286 Alloy)	9	0.0058	0.00519	12.3%	0.00020	0.0012	0.00055	0.0032
ARMI26C (A286 Alloy)	9	0.0077	0.0074	3.6%	0.00015	0.0012	0.00043	0.0034
A286-48 (A286 Alloy)	7	0.00063			0.00005	0.00065	0.00013	0.0018
Brammer BS200-4	8	0.0034	0.0037	-8.0%	0.00004	0.0022	0.00010	0.0061

[A]The average of the laboratories' calculated averages.

TABLE 4 Carbon (mass fraction %)

Note—Data from up to N=10 datasets utilized.

Material	N	Average[A] $\bar{x}$	Certified or Reference Value	Bias %	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
WT48 (718 Alloy)	10	0.0242			0.0015	0.0031	0.0041	0.0088
WN53 (718 Alloy)	9	0.0095			0.00077	0.0024	0.0022	0.0067
NIST1249 (718 Alloy)	10	0.0380			0.0014	0.0026	0.0040	0.0074
NIST1244 (600 Alloy)	9	0.0625	0.063	-0.8%	0.0016	0.0040	0.0046	0.011
Brammer BS600-4 (600 Alloy)	9	0.0345	0.034	1.4%	0.00084	0.0024	0.0024	0.0068
WT71 (600 Alloy)	6	0.0085			0.0011	0.0050	0.0030	0.014
WASP79 (Waspaloy)	7	0.0056			0.00081	0.0033	0.0023	0.0093
NIST1243 (Waspaloy)	9	0.0246			0.00094	0.0022	0.0026	0.0063
ARMI62B (Waspaloy)	9	0.0277	0.028	-1.0%	0.00062	0.0023	0.0018	0.0064
NIST1230 (A286 Alloy)	9	0.0437	0.0428	2.0%	0.0022	0.0071	0.0062	0.020
ARMI26C (A286 Alloy)	9	0.0307	0.028	9.5%	0.0010	0.0049	0.0028	0.014
A286-48 (A286 Alloy)	8	0.0261			0.00090	0.0058	0.0025	0.016
Brammer BS200-4	7	0.1066	0.107	-0.4%	0.00094	0.0053	0.0026	0.015

[A]The average of the laboratories' calculated averages.

18.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on an identical test material would, in the normal and correct operation of the test method, exceed the following values only in one instance in 20.