Designation: E1834 – 11

Standard Test Method for Analysis of Nickel Alloys by Graphite Furnace Atomic Absorption Spectrometry[1]

1. Scope

1.1 This test method describes the graphite furnace atomic absorption spectrometric analysis of nickel, such as specified by ASTM Committee B02, and having chemical compositions within the following limits:

Element	Application Range (Wt. %)
Aluminum	0. 01 - 6.00
Boron	0. 01 - 0.10
Carbon	0. 01 - 0.15
Chromium	0. 01 - 33.00
Copper	0.01 - 35.00
Cobalt	0. 01 - 20.00
Iron	0.05 - 50.00
Magnesium	0. 01 - 0.020
Molybdenum	0. 01 - 30.0
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
Tungsten	0.01 - 5.0
Vanadium	0.0005 - 1.0

1.2 The following elements may be determined using this test method:

Element	Quantification Range (µg/g)
Bismuth	0.2 - 3
Lead	0.6 - 12
Selenium	0.7 - 10
Tellurium	0.4 - 6

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 concentration ranges provided that a test method validation study that includes an instrument performance evaluation as described in Practice E1770 is performed. Additionally, the validation study shall 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 as to the intended purpose of the analytical results.

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 statements see Note 2 and Section 9.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

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

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis

E1770 Practice for Optimization of Electrothermal Atomic Absorption Spectrometric Equipment

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

2.2 *ISO Standards:*[3]

ISO Guide 31 Contents of certificates of reference materials

ISO Guide 34 Quality system guidelines for the production of reference materials

ISO Guide 98-3 Uncertainty of measurement Part 3: Guide to the expression of uncertainty in measurement (GUM:1995) – 1st Ed.

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[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.

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

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 graphite furnace atomic absorption spectrometry.

5. Significance and Use

5.1 This test method is primarily intended to test material for compliance with specifications such as those under the jurisdiction of ASTM Technical Committee B02 on Nonferrous Metals and Alloys. It may also be used to test compliance with other specifications that are compatible with the test method.

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

5.3 This is a performance-based 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 shall prepare their own work instructions. These work instructions shall include detailed operating instructions for the specific laboratory, the specific reference materials employed, and the performance acceptance criteria. It is also expected that, when applicable, each laboratory shall participate in proficiency test programs, such as described in Practice E2027, and that the results from the participating laboratory shall be satisfactory.

6. Interferences

6.1 The narrow bandwidth emitted by the source lamp makes spectral overlaps rare. However, molecular absorption bands are more likely to overlap the atomic absorption line. This problem is commonly encountered in complex nickel alloys and a background correction technique shall be employed. The use of the Zeeman background correction technique should be used in performance of this test method.

6.2 When Zeeman background correction is used for nickel alloy analysis, background absorbance up to approximately 1.5 absorbance units is adequately corrected for. The user is cautioned to examine calibration and sample solution background levels during method validation to verify that background absorbance is less than 1.5 absorbance units.

6.3 One significant problem may be encountered for determination of bismuth in alloys with iron in excess of 10 %. It is possible that use of Zeeman background correction will cause over-correction for background, resulting in erroneously low results. This potential problem results from reading the shifted pi (π) absorption components of the iron 222.9 nm line during the background read cycle. All modern spectrometers and those with chart recorders allow inspection of absorption profiles obtained during analysis. During initial instrument optimization and method validation, the user of this test method shall assess the effect of this possible issue on the results to be reported. Adjustment of char time and temperatures may help minimize this problem. Bismuth determinations made using the peak height measurement mode may also help minimize the error associated with this issue.

6.4 The atomic lines in Table 1 have been used to analyze the listed elements in nickel alloys and are suggested for the user. The user may choose to use different atomic lines provided that sensitivity is adequate. It is recommended that once atomic lines are determined, the user of this test method specify this information or reference instrument programs that include this information in their laboratory analysis procedures.

7. Apparatus

7.1 *Graphite Furnace Atomic Absorption Spectrometer,* preferably equipped with a Zeeman background correction accessory. Suitability of the spectrometer shall be established using the performance criteria described in 12.7.

7.2 *Graphite Tubes* used in this test method shall utilize a L'vov type platform.

7.3 *Sample Preparation Equipment*—Machine tools used in this test method shall be capable of removing surface oxides and other contamination from the as-received sample and then taking uncontaminated and chemically representative chips suitable for analysis.

7.4 All labware used in this test method shall be suitably cleaned for trace level 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.[4] However, the purity of acid reagents utilized in this procedure shall be suitable for trace metal analysis and shall not contain impurities in any significant amount. 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 *Reagent Water*—The purity of reagent water shall conform to the requirements of Specification D1193 for reagent water, Type I. The water purification method used shall

TABLE 1 Suggested Atomic Absorption Analytical Lines/ Interference/Modifiers

Element	Wavelength (nm)	Potential Interference
Bismuth	223.1	Fe
Lead	283.3	
Selenium	196.0	
Tellurium	214.3	

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[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. 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.

be capable of removing all elements in concentrations that might bias the test results.

8.1.3 *1000 µg/mL Palladium + 500 µg/mL Magnesium Matrix Modifier*—To prepare this modifier, mix 1 mL of 2000 µg/mL palladium (in dilute nitric acid) and 1 mL of 1000 µg/mL magnesium (in dilute nitric acid). Five µL of this solution adds 5 µg of palladium and 2.5 µg of magnesium nitrate to the furnace.

8.1.4 *$HNO_3 + HF + H_2O$ (1 + 1 + 1)*—To 150 mL of water carefully add 150 mL of HNO_3 and 150 mL HF. Mix and store in an HF resistant bottle.

8.1.5 *High Purity Nickel*—The nickel selected shall be free of the scoped analytes.

8.2 *Calibration Solutions:*

8.2.1 In this test method, calibration is based either on laboratory-prepared, pure nickel matrix-matched solutions, dissolved solid certified reference material (CRM) solutions, or on samples to which methods of standard additions spikes have been made. The matrix-matched solutions are prepared with nickel of known purity. These matrix solutions are then spiked with aliquots of single element (CRM) solutions, which contain the elements of interest. The CRMs shall be compliant with ISO Guide 31 and ISO Guide 34.

8.2.2 Sections 8.2.3–8.2.5 describe the preparation of matrix-matched calibration solutions for analysis of sample solutions that contain 1 g alloy/50 mL final dilution. It is acceptable to vary final concentrations as long as the user's method demonstrates acceptable measurement variability and detection limit (see 12.7). Section 8.2.6 describes preparation of dissolved solid CRM solutions. Section 8.2.7 describes preparation of calibration solutions for methods of additions.

8.2.3 Determine the number and composition of calibration solutions needed to cover the concentration range for each element. It is suggested that the calibration solutions have their highest concentration slightly above the highest expected sample concentration (Sk as described in Practice E1770), a concentration in the mid range of the expected sample concentrations, a concentration at or near the reporting limit (S1 as described Practice E1770), and a blank (S0 as described in Practice E1770). In any case, a minimum of three solutions including a blank shall be used for calibration. It is important that the higher concentration solutions do not yield absorbances outside of the linear range (see 12.10 to 12.11).

8.2.4 Prepare matrix solutions as follows:

NOTE 1—The following preparation instructions are written for hot plate digestion. Alternative digestion methods such as microwave or bomb digestion may be used.

8.2.4.1 Weigh 1.0 g of pure nickel into an HF resistant digestion vessel. Use one vessel for each calibration solution.

8.2.4.2 Dissolve the pure nickel in 20 mL of HNO_3 + HF + H20 (1 + 1 + 1) per gram of sample.

NOTE 2—**Caution**—If powdered nickel is used, add the acid cautiously as powdered metals tend to be very reactive.

8.2.4.3 Heat the digestion vessels gently until the nickel dissolves. Remove the beakers from the heat. Continue to heat the vessels gently to reduce this solution to approximately 5 mL in order to remove excess HF. Wet salts may form.

8.2.4.4 Cool the beakers slightly and then redissolve the salts by heating in approximately 20 mL of water.

8.2.4.5 Cool the nickel solutions and transfer into 50 mL plastic flasks. Polypropylene or polymethylpentene flasks are acceptable for this purpose.

8.2.5 Add the required amount of single element CRM solutions into the flasks, making sure to leave one analyte-free for use as a blank.

8.2.6 The laboratory may choose to prepare calibration solutions by dissolving nickel alloy certified reference materials containing analytes covering the expected sample concentration range. In this case, the calibration blank may be either a reagent blank of the reagents used for sample dissolution as described in Section 13 or a pure nickel matrix blank prepared as described in 8.2.4.

8.2.7 The method of standard additions is also an acceptable method of calibration solution preparation. Two different approaches may be used in the preparation of the spiked calibration solutions. If the method of standard additions is used it will be necessary to demonstrate instrument performance as described in Sections 12.7 through 12.10 prior to routinely preparing method of additions calibration solutions. The methods of additions calibration solutions prepared shall yield a linear instrument response when analyzed.

8.2.7.1 *Method 1:*

(1) GFAA is typically employed for the scoped elements to verify the absence of the element of interest from the material. Single point method of additions is employed by some laboratories in order to verify the absence of the analyte of interest. This approach to calibration shall be well validated as described in Section 15.

(2) Prepare one sample solution in accordance with Section 13. This solution shall serve as the unspiked calibration solution.

(3) Prepare another sample solution in accordance with Section 13. Spike the solution with an aliquot of CRM solution to yield a final solution concentration known to fall within the linear range for the analyte of interest. It is acceptable to use the solution prepared in 8.2.7.1*(2)* and make the CRM solution spike directly to the platform in the graphite tube to yield the spiked calibration solution.

8.2.7.2 *Method 2:*

(1) If samples routinely have concentrations falling above the method scope minimum, then Method 1 cannot be used. A more rigorous approach to generating the method of additions calibration solutions shall be taken.

(2) Prepare four sample solutions as described in Section 13. Spike these sample solutions with CRM aliquots to produce solutions that cover the linear range for the analyte of interest. Acceptable linear range is discussed in Section 12.

(3) It is acceptable to prepare a single sample solution as described in Section 13 and spike the sample solution directly on the furnace platform to produce the four spike calibration solutions.

8.3 *Other Materials:*

8.3.1 *Argon*—The atomic absorption spectrometer shall use argon to protect the tube from oxidation during heating and to remove vapor from the tube. The purity of the argon supply shall be as specified by the instrument manufacturer.

8.3.2 *Control Materials:*

8.3.2.1 A laboratory may choose to procure, produce, or have manufactured a chip material containing analyte contents in the range of typical samples to be used as a control material. These chips should be well blended and checked for homogeneity.

8.3.2.2 A laboratory may find it difficult to procure or have manufactured the materials described in 8.3.2.1 for all of the necessary analytes or alloys. If this is the case, then it is acceptable to prepare equivalent reference material solutions using an alternative source of nickel for the matrix solution and spiked with different single element CRM solutions.

9. Hazards

9.1 This test method involves the use of concentrated HF. Read and follow label precautions, MSDS information, and refer to Practice E50. For precautions to be observed in the use of certain other reagents in this test method, refer to Practice E50.

10. Sampling, Test Specimens, and Test Units

10.1 Laboratories shall follow written practices for sampling and preparation of test samples. These practices shall meet all customer requirements. Practices E55 and E88 also provide guidance for sampling.

11. Preparation of Apparatus

11.1 Analytical instrumentation and sample preparation equipment shall be installed and operated in a manner consistent with manufacturer's recommendations.

12. Calibration

12.1 Set up the instrument for calibration in a manner consistent with the manufacturer's recommendations. Practice E1770 provides some guidance on parameters that should be considered during set up.

12.2 Specify calibration units consistent with the concentrations of the calibration solutions prepared in 8.2. The user may choose to specify units in the atomic absorption instrument software as a mass-fraction, such as % or mg/kg, in order to simplify calculation and reporting of final results.

12.3 Define the number of replicate measurements to be made and averaged for a single reported result. Typically, a minimum of two replicates is specified.

12.4 Use of a mixed 5 µg palladium plus 2.5 µg magnesium matrix modifier is recommended. Set up the instrument autosampler to add the matrix modifier to the furnace during the heating parameter optimization study, during calibration and during sample analysis. The user may choose not to use a modifier or to use different modifiers or modifier masses, provided method validation in accordance with Section 15 demonstrates the acceptability of the selected protocol.

12.5 The manufacturer shall specify the maximum total volume of sample and modifier solution that may be pipetted onto the L'vov platform. This volume shall be taken into consideration when optimizing heating parameters.

12.6 Optimize the heating parameters to minimize sample backgrounds while maximizing absorbance. Practice E1770 provides a suggested procedure for optimization of heating parameters.

12.7 Determine instrument performance data as described in Practice E1770. Evaluate the data as follows:

12.7.1 Compare the calculated instrument detection limit to the method lower scope limit. If the instrument does not demonstrate a detection limit equivalent to or better than the lower scope limit, it is possible that the user may not be able to routinely meet this method's lower scope.

12.7.2 Compare the standard deviation determined for the most concentrated solution (S_k) with the method minimum *SD* (S_m) for a material analyzed in the interlaboratory study, which yielded a similar solution concentration. Refer to Tables 2–5 for the method minimum *SD* (S_m) data. The standard deviation should be similar if the user's instrument is performing acceptably.

12.8 Once it has been demonstrated that the instrument has acceptable measurement variability and detection limit for the elements being determined, it will not be necessary to routinely determine these attributes prior to calibration.

12.9 Follow 12.10 to calibrate using nickel matrix matched or dissolved solid CRM calibration solutions. Follow 12.11 to calibrate using the method of additions calibration solutions.

12.10 *Nickel Matrix-Matched/Solid CRM Solution Calibration:*

12.10.1 Use the blank (S_0) to zero the instrument.

12.10.2 Calibrate the instrument using the remainder of the calibration solutions ($S_1 - S_k$).

12.10.3 The calibration calculation method used should be a linear function with a zero intercept.

12.10.4 If two point calibration using a blank (S_0) and a high calibration solution (S_k), then calibration linearity shall be verified by analyzing the other calibration solutions.

12.10.5 Calculate a calibration curve using the instrument software in units of absorbance versus calibration solution concentration. The calculated calibration curve shall be linear and is described by the following equation:

$$y = mx + b \tag{1}$$

where:

y = absorbance of the solution analyzed,
m = slope of the calibration curve,
x = the concentration of the solution analyzed, and
b = y intercept of the calibration curve.

12.10.6 Linearity of the calibration curve shall be verified. Typical atomic absorption instrument software will calculate a correlation coefficient for each calibration curve. It is acceptable to rely upon the correlation coefficient as a demonstration of acceptability of calibration fit. This coefficient should routinely be at least 0.95. The correlation coefficient (r) is calculated by the following equation:

$$r = \frac{\sum_{i=1}^{n} XiYi - \sum_{i=1}^{n} Xi \sum_{i=1}^{n} Yi}{\sqrt{n \sum_{i=1}^{n} X\,i^2 - \left(\sum_{i=1}^{n} Xi\right)^2} \cdot \sqrt{n \sum_{i=1}^{n} yi^2 - \left(\sum_{i=1}^{n} yi\right)^2}} \quad (2)$$

where:

X = concentration,

y = absorbance,

n = number of standards including the blank.

The user is cautioned that when using this method it is possible to have a correlation coefficient of 1.0 and still have points that are not on the curve.

12.10.7 If two point calibration with additional linearity checks is used or if the instrument software does not calculate a correlation coefficient, then linearity shall be determined as described in Practice E1770. The minimum ratio should routinely be at least 0.95.

12.11 *Method of Standard Additions Calibration:*

12.11.1 *Method 1, Method of Additions Calibration Solutions:*

12.11.1.1 Analyze the unspiked sample calibration solution to obtain absorbance.

12.11.1.2 Analyze the spiked sample calibration solution to obtain absorbance. It is acceptable to prepare this solution directly on the tube platform using the autosampler.

12.11.1.3 Proceed directly to Section 16 in order to calculate an analysis result from the data obtained.

12.11.2 *Method 2, Method of Additions Calibration Solutions:*

12.11.2.1 Analyze each method of additions calibration solution to obtain absorbance. It is acceptable to prepare these solutions directly on the tube platform using the autosampler.

12.11.2.2 Calculate a calibration curve using the instrument software in units of absorbance versus added analyte concentration. The calculated calibration curve shall be linear and is described by the following equation:

$$y = mx + b \quad (3)$$

where:

y = absorbance of the solution analyzed,

m = slope of the calibration curve,

x = spike concentration of the solution analyzed, and

b = y intercept of the calibration curve.

12.11.2.3 Linearity of the calibration curve shall be verified. Typical atomic absorption instrument software will calculate a correlation coefficient for each calibration curve. It is acceptable to rely upon the correlation coefficient as a demonstration of acceptability of calibration fit. This coefficient should routinely be at least 0.95. See Eq 2 for calculation of correlation coefficient (r).

13. Procedure

NOTE 3—The following preparation instructions are written for hot plate digestion. Alternative digestion methods such as microwave or bomb digestion may be used.

13.1 Weigh a sample, consistent with the sample size selected for use in preparing calibration solutions, to the nearest 0.0001 g and place it into a HF resistant digestion vessel.

13.2 Add 20 mL of $HNO_3 + HF + H_2O$ (1 + 1 + 1) per gram of sample.

13.3 Heat the digestion vessel gently until the sample is dissolved and the solution volume is reduced to approximately 5 mL. Wet salts may form.

13.4 Remove the digestion vessel from the heat and cool slightly. Add approximately 20 mL of water.

13.5 Heat the sample solution until any wet salts are dissolved.

13.6 Cool the solution and transfer into a plastic volumetric flask. The volume of this flask shall be consistent with the final dilution volume of the calibration solutions.

13.7 Dilute the flask to volume and mix well.

13.8 Analyze the sample solution in accordance with the instrument manufacturer's instructions and the laboratory's standard operating procedure, using the calibration generated in Section 15. The same modifier and modifier mass introduced during calibration shall be used for sample analysis.

13.9 If the method of standard additions is being used the appropriate CRM solution may be spiked to the sample directly on the tube platform using the autosampler.

13.10 Analyze a control sample periodically throughout the run of the batch and at the end of the run. Use the control sample to evaluate the need for recalibration and reanalysis of samples. Refer to Section 14 for specific information on control sample analysis.

14. Control

14.1 The laboratory shall establish procedures for calibration curve drift control. One suggested method involves the use of a control chart to monitor drift. Prepare a control chart for each control sample. Refer to Practice E1329 for guidance on use of control charts. Users of this test method are discouraged from using certified reference materials as routine control materials.

14.2 Most atomic absorption instrument manufacturer's software allows the use of programmable control sample tolerances. It is acceptable to calculate control limits and to use these as limits in the instrument software.

14.3 The individual laboratories' analysis procedures will typically specify reanalysis of affected samples if control samples indicate that the calibration is no longer valid.

15. Method Validation

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

15.2 Initially, the laboratory should prepare and analyze solid CRMs and/or RMs using this method to obtain these data.

If there are no solid CRMs or RMs available for the alloys/analytes being determined, then spike recovery studies using alloy samples should be part of the validation process. The precision and bias data obtained for these materials should be compared to the precision and bias data stated in this method.

15.3 Any laboratory demonstrating significantly worse precision and bias data should attempt to identify and correct any problems associated with their application of this test method.

15.4 The user of this test method shall weigh customer requirements and the laboratory's data quality objectives and justify acceptance of the method validation data.

15.5 The test method validation study shall be documented.

16. Calculation

16.1 *Calculation if Calibration via Nickel Matrix-Matched or Dissolved Solid CRM Solutions*—The calibration function shall yield a linear plot described by the following:

16.1.1 If the user chooses to specify calibration units in the atomic absorption instrument software to express the concentration of analyte contained in the sample as a mass fraction, then no other calculations other than sample weight correction is required. Results may be taken directly from the instrument readout.

16.1.2 If the user specified analyte concentration as a volume-fraction into the software, it will be necessary to convert the analyte volume-fraction concentrations obtained for the sample solution into analyte mass-fraction concentrations contained in the sample. For example, if the sample is prepared as 1 g of sample diluted to a final volume of 50 mL solution, an analyte solution concentration of 0.1 µg analyte/mL of solution corresponds to 5 µg/g (m/m) analyte in the sample.

16.2 *Calculation Using Method 1, Method of Standard Additions*—

16.2.1 Calculate the analyte concentration of the original sample solution using the following formula:

$$\textit{unknown concentration} = \frac{(\textit{spike concentration})\cdot(\textit{sample absorbance})}{(\textit{spike absorbance}) - (\textit{sample absorbance})} \quad (4)$$

unknown concentration = analyte concentration in the unknown sample solution,
spike concentration = analyte spike concentration added to the unknown sample solution,
spike absorbance = absorbance obtained for the spiked sample solution, and
sample absorbance = absorbance obtained for the unspiked sample solution.

16.3 *Calculation Using Method 2, Method of Standard Additions:*

16.3.1 The *y*-intercept (*b*) of the calibration curve calculated for the method of standard additions (see 12.11.2) is the analyte concentration of the unknown sample. Report this value without further calculation.

17. Report

17.1 Results shall be reported in a manner consistent with customer requirements. When uncertainty estimates are required, results should be reported in accordance with the guidance provided in the ISO Guide 98-3. In this document, it is explained that the analyst shall 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 1 *s* (one standard deviation), and *k* is an expansion factor typically chosen as $k = 2$ to approximate a 95 % level of confidence. It is expected that the laboratory shall 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 decimal places.

18. Precision and Bias

18.1 The precision of this test method is based on an interlaboratory study conducted in 2010. Eight analysts participated in this study, analyzing six materials, to determine the concentration of four different elements. Each test result reported represents the average of three individual determinations and all participants were instructed to report three replicate test results for each element. Practice E691 was followed for the design and analysis of the data; the details are given in RR:E01-1117.[5]

18.1.1 *Repeatability Limit, r*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the *r* value for that material; *r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

18.1.1.1 Repeatability limits are listed in Tables 2-5.

18.1.2 *Reproducibility Limit, R*—Two test results shall be judged not equivalent if they differ by more than the *R* value for that material; *R* is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

18.1.2.1 Reproducibility limits are listed in Tables 2-5.

18.1.3 The above terms (*repeatability limit* and *reproducibility limit*) are used as specified in Practice E177.

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1117.

TABLE 2 Lead (mg/kg)

Material	Average, $\bar{\chi}^{A}$	Certified Value^{B}	Average Recovery, %	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
RM WR01 Millings (718 Alloy)	4.94			0.11	0.41	0.31	1.15
RM WP82 Millings (718 Alloy)	0.45			0.06	0.11	0.16	0.31
NIST SRM 897 Millings (Nickel High Temperature Alloy)	11.27	11.7	96.32	0.41	0.76	1.14	2.13
NIST SRM 898 Millings (Nickel High Temperature Alloy)	2.00	2.5	80.00	0.06	0.21	0.18	0.58
NIST SRM 899 Millings (Nickel High Temperature Alloy)	3.93	3.9	100.77	0.14	0.43	0.39	1.21
ARMI IARM 189A Millings (Pure Nickel)	2.95	2.9	101.72	0.11	0.27	0.30	0.75

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

[B] Certified values reported by NIST and ARMI, respectively.

TABLE 3 Selenium (mg/kg)

Material	Average, $\bar{\chi}^{A}$	Certified Value^{B}	Average Recovery, %	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
RM WR01 Millings (718 Alloy)	3.20			0.14	0.38	0.40	1.06
RM WP82 Millings (718 Alloy)	0.40			0.06	0.13	0.17	0.37
NIST SRM 897 Millings (Nickel High Temperature Alloy)	9.26	9.1	101.76	0.37	0.79	1.05	2.23
NIST SRM 898 Millings (Nickel High Temperature Alloy)	2.03	2.0	101.50	0.20	0.28	0.55	0.77
NIST SRM 899 Millings (Nickel High Temperature Alloy)	9.30	9.5	97.89	0.34	0.78	0.96	2.18
ARMI IARM 189A Millings (Pure Nickel)	2.67	2.1	127.14	0.29	0.46	0.81	1.28

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

[B] Certified values reported by NIST and ARMI, respectively.

TABLE 4 Bismuth (mg/kg)

Material	Average, $\bar{\chi}$[A]	Certified Value[B]	Average Recovery, %	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
RM WR01 Millings (718 Alloy)	3.00			0.14	0.44	0.40	1.23
RM WP82 Millings (718 Alloy)	0.21			0.05	0.06	0.13	0.17
NIST SRM 897 Millings (Nickel High Temperature Alloy)	0.57			0.03	0.12	0.08	0.34
NIST SRM 898 Millings (Nickel High Temperature Alloy)	1.07			0.05	0.14	0.13	0.40
NIST SRM 899 Millings (Nickel High Temperature Alloy)	0.27			0.03	0.04	0.10	0.11
ARMI IARM 189A Millings (Pure Nickel)	2.98	2.6	114.62	0.19	0.46	0.52	1.28

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

[B] Certified values reported by NIST and ARMI, respectively.

TABLE 5 Tellurium (mg/kg)

Material	Average, $\bar{\chi}$[A]	Certified Value[B]	Average Recovery, %	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
RM WR01 Millings (718 Alloy)	2.85			0.07	0.40	0.20	1.11
RM WP82 Millings (718 Alloy)	0.56			0.05	0.29	0.14	0.80
NIST SRM 897 Millings (Nickel High Temperature Alloy)	1.07	1.05	101.90	0.05	0.18	0.15	0.50
NIST SRM 898 Millings (Nickel High Temperature Alloy)	0.64	0.54	118.52	0.04	0.08	0.11	0.22
NIST SRM 899 Millings (Nickel High Temperature Alloy)	5.68	5.9	96.27	0.21	0.57	0.60	1.58
ARMI IARM 189A Millings (Pure Nickel)	2.11	1.7	124.12	0.09	0.57	0.25	1.60

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

[B] Certified values reported by NIST and ARMI, respectively.

18.1.4 Any judgment in accordance with statements 18.1.1 and would have an approximate 95% probability of being correct.

18.2 *Bias*—Four certified reference materials were included in the interlaboratory study. The average recoveries, as reported by the participating laboratoriess, can be found in Tables 2-5.

18.3 The precision statement was determined through statistical examination of 534 test results, reported by eight individuals, on six alloys (as specified in Tables 2-5).

18.4 The reference materials used in this study were provided by the following organizations:

WP82, WR01- ATI Allvac
SRMs 897, 898, 899- National Institute of Standards and Technology
IARM 189A- Analytical Reference Materials International

19. Keywords

19.1 atomic absorption spectrometry; GF-AAS; graphite furnace; nickel; nickel alloys

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