Designation: E2823 – 17

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

This standard is issued under the fixed designation E2823; 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 mass spectrometric analysis of nickel and nickel allys, as specified by Committee B02, and having chemical compositions within the following limits:

| Element | Application Range (Mass Fraction %) |
|---|---|
| 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 method.

| Element | Quantification Range (µg/g) |
|---|---|
| Antimony | 0.5–50 |
| Bismuth | 0.1–11 |
| Gallium | 2.9–54 |
| Lead | 0.4–21 |
| Silver | 1–35 |
| Tin | 2.2–97 |
| Thallium | 0.5–3.0 |

1.3 This method has only been interlaboratory tested for the elements and ranges specified. It may be possible to extend this method to other elements or different composition ranges provided that method validation that includes evaluation of method sensitivity, precision, and bias as described in this document is performed. Additionally, the validation study must evaluate the acceptability of sample preparation methodology using reference materials and/or spike recoveries. The user is cautioned to carefully evaluate the validation data as to the intended purpose of the analytical results. Guide E2857 provides additional guidance on method validation.

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.* Specific safety hazard statements are given in Section 9.

## 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

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

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

[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.

E2165 Practice for Establishing an Uncertainty Budget for the Chemical Analysis of Metals, Ores, and Related Materials (Withdrawn 2007)[3]
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 17025 General Requirements for the Competence of Calibration and Testing Laboratories
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 mass spectrometry.

## 5. Significance and Use

5.1 This test method for the chemical analysis of nickel and 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 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 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 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 When possible, analyte isotopes are selected that are free from mass overlap interferences. Because isotope choices are limited, this is not always an option. It is the responsibility of the user to determine run conditions and parameters that avoid or compensate for interferences that may bias test results.

6.2 The use of an internal standard may compensate for the physical interferences resulting from variations in sample and calibration solution aerosol transport rates. The user may chose to add the internal standard by spiking each solution with a specified amount of an appropriate certified reference material (CRM) solution. Alternatively, on-line addition of a peripheral internal standard solution during sample analysis is also possible provided acceptable instrument sensitivity is maintained.

6.3 Isobaric and polyatomic mass overlap interferences are best addressed by selecting an alternate atomic mass. Some instrument manufacturers offer software options for mathematically correcting for common interferences, but the user is cautioned to carefully evaluate this approach to mass overlap correction. However, some laboratories participating in the interlaboratory study found it necessary to generate a mathematical correction for the effect of the ZrO interference on the Ag 107 isotope. In this case the Zr 91 isotope was used for zirconium determination.

6.4 Modern instruments may have a collision or reaction cell that can use ion-molecule collisions or reactions to remove spectral interferences. The user of this method must examine this information to ascertain the need for collision/reaction cells for the removal of spectral interferences. However, it should be noted that no collision/reaction gases were used by the laboratories participating in the interlaboratory study of the elements listed in the Scope, thus implying that the use of collision/reaction gases is not required for determination of those elements.

6.5 The isotopes listed 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 multiple isotopes to help verify that atomic mass selection is optimized for the particular alloy being determined. It is recommended that once isotopes and appropriate spectral corrections are determined, the user of this method specify this information or reference instrument programs, which include this information in their laboratory analysis procedures.

## 7. Apparatus

7.1 Suitability of an Inductively Coupled Plasma Mass Spectrometer for testing of this method will be established using the performance criteria described in section 12.1. The sample introduction system shall be capable of handling solutions containing trace amounts of HF. Each instrument shall be installed and operated according to the manufacturer's recommendations.

**TABLE 1 Suggested Isotopes/Interference**

| Element | Isotope | Potential Interference |
|---|---|---|
| Antimony | 121 | |
| Bismuth | 209 | |
| Gallium | 71 | |
| Lead | 208 | |
| Silver | 107 | ZrO, FeCr |
| Tin | 120 | MoO |
| Thallium | 205 | |

[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.

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

7.3 All labware used should 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.[5] However, the purity of acid reagents utilized in this procedure shall be suitable for trace metal analysis and should not contain any significant amount of the analyte. 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*—The purity of water used in this test method shall conform to the requirements of Specification D1193 for reagent water, Type I. 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 recommended. The use of an internal standard may compensate for the physical interferences resulting from variations in sample and calibration solution aerosol transport rates. Select an internal standard element of similar atomic mass to the analyte and one that is not commonly found in the samples to be determined. The exact concentration added is not critical, however, the amount added should yield a significant signal when measured.

8.2 *Calibration Solutions:*

8.2.1 In this test method, calibration is based on laboratory-prepared, pure nickel matrix- matched solutions. The matrix solutions are prepared with nickel of known purity. These matrix solutions are then spiked with aliquots of single element certified reference material (CRM) solutions which contain the elements of interest.

8.2.2 Step 8.2.3 and following describe the preparation of calibration solutions for analysis of sample solutions that contain 1 g alloy/L final dilution. It is acceptable to vary final concentrations as long as the user's method demonstrates adequate sensitivity and precision (see 12.1).

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, a concentration in the mid-range of the expected sample concentrations, a concentration at or near the reporting limit, and a blank. In any case, a minimum of three solutions including a blank must be used for calibration.

8.2.4 Prepare matrix solutions as follows:

8.2.4.1 Weigh 0.5 g of pure nickel into an HF resistant digestion vessel. Use one vessel for each calibration solution to be made. Note that using 0.5 g of nickel approximates the mass fraction of nickel (50 %) found in 1 g of a typical nickel alloy.

8.2.4.2 Dissolve the pure nickel in 20 mL of acid mixture per gram of sample. Select acid mixtures that will dissolve the alloys to be analyzed using this method.

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

8.2.4.3 A mixture of HCl + $HNO_3$ (9 + 1), HCl + $H_2O$ + $HNO_3$ (3 + 2 + 1), or $HNO_3$ + HF + $H_2O$ (1 + 1 + 1) will dissolve many types of nickel alloys . For high Mo-Cr alloys it has been found that concentrated HCl with the addition of concentrated $HNO_3$ dropwise may be necessary to avoid passivation.

8.2.4.4 Heat the digestion vessels gently until the nickel dissolves. Remove the beakers from the heat, add 10 drops of 49 % HF, and swirl gently. If $HNO_3$ + HF + $H_2O$ (1 + 1 + 1) is used for digestion, it is not necessary to add additional HF. The laboratory may choose to reduce this solution to wet salts in order to remove excess HF and then re-dissolve by heating the salts in approximately 20 mL of water.

8.2.4.5 If an internal standard is used, add the predetermined amount into each solution.

8.2.4.6 Cool the nickel solutions and transfer into 1-L plastic flasks. Polypropylene or polymethylpentene flasks are acceptable for this purpose.

8.2.5 Add the needed amount of single element CRM solutions to the flasks, ensuring to leave one analyte-free for use as a blank. Maintain the acidity necessary to assure solution stability. The acidity given on the solution CRM certificate of analysis will provide guidance on the necessary acid concentrations needed to do this. Typically, if these solutions are to match samples prepared using one gram of alloy diluted to1-L, the quantity of acids used in 8.2.4 will be sufficient to hold all analytes in solution.

8.3 *Other Materials:*

8.3.1 *Argon*—The ICP-MS argon supply should be in accordance with the recommendations of 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. Additional guidance on the production of these control materials may be found in Guide E2972.

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.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. https://.acs.pubs.org/reagents/index.html. 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, https://www.usp.org.

8.3.3 *Collision/Reaction Gases*—Collision and/or reaction gases may be used to minimize the effects of isobaric and polyatomic mass interferences. Manufacturers typically will provide guidance upon the type and purity of collision and reaction gases to be used for a specific analyte.

## 9. Hazards

9.1 This method involves the use of HF. Read and follow label precautions, SDS 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.

10.2 Test specimens should be obtained by milling or drilling chips that are clean and of sufficient quantity to fulfill the sample mass required by the procedure.

## 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 It will be necessary to establish that the instrument being used is capable of demonstrating acceptable sensitivity and precision for the elements being determined. Once it has been demonstrated that the instrument has acceptable sensitivity and precision for these elements, it will not be necessary to routinely evaluate sensitivity and precision. Evaluate equipment sensitivity and precision as described in sections 12.1.1 and 12.1.2.

12.1.1 *Sensitivity*—Sensitivity shall be evaluated by first establishing a calibration curve for each element being determined using calibration solutions prepared as described in section 8.2. At a minimum the calibration curve will contain two points. After thorough rinsing, the blank solution is analyzed 10 times. Calculate 3 times the standard deviation of this determination as an approximation of the limit of detection . Calculate 10 times the standard deviation to approximate the limit of quantification. If the instrument/parameter selection of the user does not produce an estimated limit of detection equal to or better than the lower scope limit of the method for the element(s) being determined, then it is probable the method user will be unable to meet the method's lower scope limit. If the instrument/parameter selection of the user does not produce a limit of quantification equal to or better than the lower scope limit of the method for the element(s) being determined, then it is possible the method user will be unable to consistently meet the method's lower scope limit.

12.1.2 *Precision*—The short-term precision shall be determined as follows. Using the same calibration generated in 12.1.1, analyze the high calibration solution 10 times using the instrument/parameters selected by the method user. Calculate the % Relative Standard Deviation (% RSD) as follows:

$$\% \text{ RSD} = \frac{100s}{\bar{C}} \tag{1}$$

where:

$s$ = estimated standard deviation, and
$\bar{C}$ = average of the 10 results obtained for the high calibration solution.

12.1.2.1 The calculated % RSD should be < 5.0 %. If it is not, the user of this method may not be able to meet the performance criteria of the method. Some factors that may affect precision for inductively coupled plasma emission spectrometers may also affect inductively coupled plasma mass spectrometers. Practice E1479 provides limited guidance as to the parameters, which may have an effect on the precision characteristics of both spectrometer types. Instrument troubleshooting manuals provided by the manufacturer of the equipment may also provide guidance for optimizing performance for the specific instrument being used.

12.2 *Calibration Procedure:*

12.2.1 Set up the instrument for calibration in a manner consistent with the manufacturer's recommendations.

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

12.2.3 Define the number of replicate measurements to be made and averaged for a single reported result. Typically, a minimum of 2 replicates is specified.

12.2.4 Calibrate the instrument using the calibration solutions. Calibration curves for ICP-MS are generally linear over several orders of magnitude. Typical calibration methods include calculation of a linear function using a calculated intercept, calculation of a linear function while forcing the intercept through zero, and calculation of a linear function using concentration weighting. Method validation per Section 15 may help the lab in selecting an appropriate calibration algorithm.

12.2.5 The user of this method must verify the quality of the calibration fit. Typical ICP-MS instrument software will calculate a correlation coefficient for each calibration curve. It is acceptable to rely upon the correlation coefficient as a demonstration of calibration fit. This coefficient should be 0.999 or better. The user of this method may choose other methods to judge the quality of a calibration fit, such as checking the residuals for trends and calculating a lack of fit parameter. If the user elects to use a linear equation with a calculated intercept then the correlation coefficient ($r$) is calculated by the following equation:

$$r = \frac{\sum_{i=1}^{n} XiYi - \sum_{i=1}^{n} Xi \sum_{i=1}^{n} Yi}{\sqrt{n \sum_{i=1}^{n} Xi^2 - \left(\sum_{i=1}^{n} Xi\right)^2} \times \sqrt{n \sum_{i=1}^{n} Yi^2 - \left(\sum_{i=1}^{n} Yi\right)^2}} \tag{2}$$

where:

$X$ = concentration,
$Y$ = intensity, and

$n$ = number of calibration solutions including the blank.

12.2.6 The user is cautioned that when using this test method it is possible to have a correlation coefficient of 1.0 and still have points that are not on the curve. Other methods exist to judge the quality of a calibration fit, including checking the residuals for trends and calculating a lack of fit parameter. The user of this test method may choose to use these methods.

## 13. Procedure

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 an HF-resistant digestion vessel.

13.2 Add the same volume and acid mixture used to prepare the calibration solutions (8.2.4) and cover.

13.3 Heat the digestion vessel gently until the sample is dissolved.

13.4 Remove the digestion vessel from the heat, add the same volume of 49 % HF used to prepare the calibration solutions, and swirl gently. If $HNO_3$ + HF + $H_2O$ (1 + 1 + 1) is used for digestion it is not necessary to add additional HF. If $HNO_3$ + HF + $H_2O$ (1 + 1 + 1) is used for digestion it may not be necessary to cover the digestion vessel. The laboratory may choose to reduce this solution to wet salts in order to remove excess HF and then re-dissolve by heating the salts in approximately 20 mL of water.

13.5 Cool the solution and transfer into a plastic volumetric flask. It is acceptable to use serial dilution of the sample solution to achieve the same sample mass-to-volume ratio and acid concentration as the calibration solutions. The mass-to-volume ratio and acid concentration of the sample solutions and calibration solutions must match.

13.6 Add an internal standard to the final dilution flask if used in the calibration solutions.

13.7 Dilute the final dilution flask to volume, and mix well.

NOTE 1—Other potential sample preparation issues that should be considered are given as follows. A validation study as specified in Section 12, should evaluate the validity of the sample preparation method.

*(1)* Rare earth elements may precipitate and the use of small amounts of HF during digestion is suggested. Method validation should be used to demonstrate the validity of the preparation method selected.

*(2)* Caution should be used in boiling solutions for the analysis of boron and silicon with HF as volatile fluorides may be lost. Sealed digestion bombs may be used where method validation dictates their use.

*(3)* Some laboratories have found that separation of the analytes of interest from the matrix is useful for analysis of analytes for which serious spectral overlaps from the matrix exist. Method validation of this approach to analysis should be performed.

13.8 Analyze the sample solution according to the instrument manufacturer's instructions and the laboratory's standard operating procedure, using the calibration generated in Section 12.

13.9 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 will 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 method are discouraged from using certified reference materials as routine control materials.

14.2 Most ICP-MS 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 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. Guide E2857 provides guidance on method validation.

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

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

15.5 The method validation study shall be documented.

## 16. Calculations

16.1 If the user chooses to specify units in the ICP-MS instrument software to express the composition of analyte contained in the sample as a mass fraction, then no other calculations other than sample weight correction will be necessary. Results may be taken directly from the instrument readout.

16.2 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 contained in the sample. For example if the sample is prepared as 1 g of sample diluted to a final volume of 1000 mL solution, an analyte solution concentration of 1.00 µg analyte/L of solution corresponds to 1 µg/g (m/m) in the sample.

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 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 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 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 will 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. A total of seven laboratories participated in this study, testing samples of eight different nickel alloys for their elemental contents. Not every laboratory was able to submit results for every alloy/element pair, however each "test result" reported represents an individual ICP-MS determination, and all participants were asked to report triplicate test results for each alloy/element combination. Practice E691 was followed for the design and analysis of the data; the details are given in RR:E01-1115[6].

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 2-8.

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 2-8.

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

18.1.4 Except in cases where fewer than six laboratories reported usable data, any judgment in accordance with statements 18.1.1 and 18.1.2 would have an approximate 95 % probability of being correct.

18.2 *Bias*—Certified values are reported in Tables 2-8, along with the relative bias of the reported average recoveries.

18.3 The precision statement was determined through statistical examination of 1185 test results, submitted by seven laboratories, measuring eight elements, in eight nickel alloys.

18.3.1 The eight alloys were described as:

Alloy 1: Nickel-Base High Temperature Alloy (Trace Alloy ″A″)
Alloy 2: Nickel-Base High Temperature Alloy (Trace Alloy ″B″)
Alloy 3: Nickel-Base High Temperature Alloy (Trace Alloy ″C″)
Alloy 4: Nickel Alloy IN 100 – 346
Alloy 5: Nickel Alloy IN – 345
Alloy 6: Nickel Alloy IN 718 - WP77
Alloy 7: Nickel Alloy IN 718 - WP82
Alloy 8: Nickel Alloy IN 718 - WR01

18.4 To judge the equivalency of two test results, it is recommended to choose the alloy material that is closest in characteristics to the test material.

## 19. Keywords

19.1 ICP-MS; inductively coupled plasma mass spectrometry; nickel; nickel alloys

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1115.

**TABLE 2 Antimony (µg/gm)**

| Material | Number of data sets contributing to precision calculations | Average[A] $\bar{x}$ | Reference Value[B] | Bias (Avg Recovery) | Repeatability Standard Deviation $s_r$ | Reproducibility Standard Deviation $s_R$ | Repeatability Limit r | Reproducibility Limit R |
|---|---|---|---|---|---|---|---|---|
| Alloy 1 | 6 | 1.597 | NA | NA | 0.081 | 0.165 | 0.226 | 0.461 |
| Alloy 2 | 6 | 1.490 | NA | NA | 0.049 | 0.167 | 0.136 | 0.469 |
| Alloy 3 | 6 | 1.536 | NA | NA | 0.058 | 0.161 | 0.163 | 0.452 |
| Alloy 4 | 7 | 49.169 | 47 | 104.6% | 0.532 | 1.931 | 1.489 | 5.407 |
| Alloy 5 | 6 | 1.887 | NA | NA | 0.026 | 0.145 | 0.073 | 0.406 |
| Alloy 6 | 7 | 6.209 | NA | NA | 0.160 | 0.370 | 0.449 | 1.037 |
| Alloy 7 | 7 | 2.248 | NA | NA | 0.075 | 0.262 | 0.210 | 0.733 |
| Alloy 8 | 7 | 0.867 | NA | NA | 0.065 | 0.095 | 0.182 | 0.266 |

[A] The average of the laboratories' calculated averages.
[B] Reference values reported by NIST for Alloys 1 through 3, and BCS for Alloys 4 and 5.

**TABLE 3 Bismuth (µg/gm)**

| Material | Number of data sets contributing to precision calculations | Average[A] $\bar{x}$ | Reference Value[B] | Bias (Avg Recovery) | Repeatability Standard Deviation $s_r$ | Reproducibility Standard Deviation $s_R$ | Repeatability Limit r | Reproducibility Limit R |
|---|---|---|---|---|---|---|---|---|
| Alloy 1 | 6 | 0.533 | NA | NA | 0.013 | 0.085 | 0.037 | 0.237 |
| Alloy 2 | 6 | 0.987 | NA | NA | 0.020 | 0.056 | 0.057 | 0.157 |
| Alloy 3 | 6 | 0.243 | NA | NA | 0.017 | 0.053 | 0.048 | 0.147 |
| Alloy 4 | 7 | 10.380 | 10.4 | 99.8% | 0.391 | 1.214 | 1.096 | 3.399 |
| Alloy 5 | 3 | 0.005 | NA | NA | 0.003 | 0.003 | 0.008 | 0.008 |
| Alloy 6 | 7 | 0.368 | NA | NA | 0.020 | 0.059 | 0.056 | 0.165 |
| Alloy 7 | 6 | 0.140 | NA | NA | 0.009 | 0.020 | 0.025 | 0.056 |
| Alloy 8 | 7 | 2.973 | NA | NA | 0.054 | 0.243 | 0.151 | 0.680 |

[A] The average of the laboratories' calculated averages.
[B] Reference values reported by NIST for Alloys 1 through 3, and BCS for Alloys 4 and 5.

**TABLE 4 Gallium (µg/gm)**

| Material | Number of data sets contributing to precision calculations | Average[A] $\bar{x}$ | Reference Value[B] | Bias (Avg Recovery) | Repeatability Standard Deviation $s_r$ | Reproducibility Standard Deviation $s_R$ | Repeatability Limit r | Reproducibility Limit R |
|---|---|---|---|---|---|---|---|---|
| Alloy 1 | 6 | 6.315 | NA | NA | 0.158 | 0.606 | 0.442 | 1.696 |
| Alloy 2 | 6 | 6.267 | NA | NA | 0.155 | 0.518 | 0.435 | 1.450 |
| Alloy 3 | 6 | 6.034 | NA | NA | 0.128 | 0.523 | 0.357 | 1.466 |
| Alloy 4 | 7 | 53.144 | 50.6 | 105.0% | 1.461 | 3.479 | 4.090 | 9.742 |
| Alloy 5 | 7 | 7.684 | 8.2 | 93.7% | 0.063 | 0.601 | 0.177 | 1.683 |
| Alloy 6 | 6 | 12.868 | NA | NA | 0.382 | 2.212 | 1.069 | 6.193 |
| Alloy 7 | 6 | 9.002 | NA | NA | 0.183 | 1.694 | 0.513 | 4.744 |
| Alloy 8 | 6 | 48.208 | NA | NA | 0.583 | 8.770 | 1.632 | 24.557 |

[A] The average of the laboratories' calculated averages.
[B] Reference values reported by NIST for Alloys 1 through 3, and BCS for Alloys 4 and 5.

**TABLE 5 Lead (µg/gm)**

| Material | Number of data sets contributing to precision calculations | Average[A] $\bar{x}$ | Reference Value[B] | Bias (Avg Recovery) | Repeatability Standard Deviation $s_r$ | Reproducibility Standard Deviation $s_R$ | Repeatability Limit r | Reproducibility Limit R |
|---|---|---|---|---|---|---|---|---|
| Alloy 1 | 7 | 11.666 | 11.7 | 99.7% | 0.177 | 1.153 | 0.496 | 3.229 |
| Alloy 2 | 7 | 2.117 | 2.5 | 84.7% | 0.045 | 0.341 | 0.125 | 0.955 |
| Alloy 3 | 7 | 4.048 | 3.9 | 103.8% | 0.156 | 0.656 | 0.437 | 1.837 |
| Alloy 4 | 7 | 20.363 | 21.0 | 97.0% | 0.348 | 1.475 | 0.975 | 4.129 |
| Alloy 5 | 6 | 0.127 | 0.21 | 60.5% | 0.012 | 0.074 | 0.033 | 0.206 |
| Alloy 6 | 7 | 1.857 | NA | NA | 0.063 | 0.139 | 0.177 | 0.389 |
| Alloy 7 | 7 | 0.490 | NA | NA | 0.037 | 0.101 | 0.104 | 0.281 |
| Alloy 8 | 7 | 5.313 | NA | NA | 0.098 | 0.452 | 0.274 | 1.265 |

[A] The average of the laboratories' calculated averages.
[B] Reference values reported by NIST for Alloys 1 through 3, and BCS for Alloys 4 and 5.

**TABLE 6 Silver (µg/gm)**

| Material | Number of data sets contributing to precision calculations | Average[A] $\bar{x}$ | Reference Value[B] | Bias (Avg Recovery) | Repeatability Standard Deviation $s_r$ | Reproducibility Standard Deviation $s_R$ | Repeatability Limit r | Reproducibility Limit R |
|---|---|---|---|---|---|---|---|---|
| Alloy 1 | 5 | 0.899 | NA | NA | 0.176 | 0.704 | 0.494 | 1.971 |
| Alloy 2 | 5 | 0.873 | NA | NA | 0.052 | 0.581 | 0.145 | 1.627 |
| Alloy 3 | 5 | 0.814 | NA | NA | 0.146 | 0.713 | 0.408 | 1.996 |
| Alloy 4 | 6 | 34.107 | 35.0 | 97.4% | 1.042 | 2.453 | 2.917 | 6.868 |
| Alloy 5 | 5 | 0.292 | NA | NA | 0.037 | 0.377 | 0.104 | 1.057 |
| Alloy 6 | 5 | 0.122 | NA | NA | 0.025 | 0.105 | 0.071 | 0.294 |
| Alloy 7 | 5 | 0.103 | NA | NA | 0.020 | 0.112 | 0.055 | 0.312 |
| Alloy 8 | 5 | 0.121 | NA | NA | 0.020 | 0.110 | 0.056 | 0.308 |

[A] The average of the laboratories' calculated averages.
[B] Reference values reported by NIST for Alloys 1 through 3, and BCS for Alloys 4 and 5.

**TABLE 7 Tin (µg/gm)**

| Material | Number of data sets contributing to precision calculations | Average[A] $\bar{x}$ | Reference Value[B] | Bias (Avg Recovery) | Repeatability Standard Deviation $s_r$ | Reproducibility Standard Deviation $s_R$ | Repeatability Limit r | Reproducibility Limit R |
|---|---|---|---|---|---|---|---|---|
| Alloy 1 | 6 | 4.193 | NA | NA | 0.073 | 0.219 | 0.205 | 0.614 |
| Alloy 2 | 6 | 4.127 | NA | NA | 0.062 | 0.397 | 0.174 | 1.112 |
| Alloy 3 | 6 | 4.159 | NA | NA | 0.116 | 0.344 | 0.325 | 0.964 |
| Alloy 4 | 7 | 96.138 | 91 | 105.6% | 0.992 | 4.492 | 2.777 | 12.577 |
| Alloy 5 | 7 | 4.462 | 5.6 | 79.7% | 0.099 | 0.433 | 0.278 | 1.212 |
| Alloy 6 | 7 | 0.769 | NA | NA | 0.064 | 0.385 | 0.178 | 1.079 |
| Alloy 7 | 7 | 1.142 | NA | NA | 0.116 | 0.504 | 0.326 | 1.410 |
| Alloy 8 | 7 | 0.914 | NA | NA | 0.122 | 0.467 | 0.343 | 1.308 |

[A] The average of the laboratories' calculated averages.
[B] Reference values reported by NIST for Alloys 1 through 3, and BCS for Alloys 4 and 5.

**TABLE 8 Thallium (µg/gm)**

| Material | Number of data sets contributing to precision calculations | Average[A] $\bar{x}$ | Reference Value[B] | Bias (Avg Recovery) | Repeatability Standard Deviation $s_r$ | Reproducibility Standard Deviation $s_R$ | Repeatability Limit r | Reproducibility Limit R |
|---|---|---|---|---|---|---|---|---|
| Alloy 1 | 7 | 0.540 | 0.51 | 105.9% | 0.035 | 0.097 | 0.099 | 0.271 |
| Alloy 2 | 6 | 2.875 | 2.75 | 104.5% | 0.054 | 0.344 | 0.153 | 0.964 |
| Alloy 3 | 7 | 0.283 | 0.252 | 112.3% | 0.022 | 0.055 | 0.061 | 0.154 |
| Alloy 4 | 7 | 1.765 | 1.8 | 98.1% | 0.034 | 0.190 | 0.096 | 0.532 |
| Alloy 5 | 4 | 0.029 | NA | NA | 0.003 | 0.052 | 0.009 | 0.147 |
| Alloy 6 | 7 | 2.161 | NA | NA | 0.048 | 0.133 | 0.136 | 0.373 |
| Alloy 7 | 7 | 0.105 | NA | NA | 0.013 | 0.018 | 0.035 | 0.050 |
| Alloy 8 | 7 | 0.646 | NA | NA | 0.016 | 0.049 | 0.045 | 0.136 |

[A] The average of the laboratories' calculated averages.
[B] Reference values reported by NIST for Alloys 1 through 3, and BCS for Alloys 4 and 5.