ASTM INTERNATIONAL

Designation: E2594 – 09 (Reapproved 2014)

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

1. Scope

1.1 This test method describes the inductively coupled plasma atomic emission spectrometric analysis of nickel alloys, such as specified by Committee B02, and having chemical compositions within the following limits:

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

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

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

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

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

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in 8.2.6.3 and safety hazard statements are given in Section 9.

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

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

2.2 *ISO Standards:*[3]

ISO/IEC 17025 General Requirements for the Competence of Calibration and Testing Laboratories

ISO Guide 31 Reference Materials—Contents of Certificates and Labels

ISO Guide 34 General Requirements for the Competence of Reference Material Producers

ISO Guide 98-3 Uncertainty of Measurement Part 3: Guide to the Expression of Uncertainty in Measurement (GUM:1995), First Edition

3. Terminology

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

4. Summary of Test Method

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

5. Significance and Use

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

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

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

6. Interferences

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

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

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

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

6.5 Table 1 suggests wavelengths that the user may use for analysis of nickel alloys. Each line was used by at least one laboratory during the interlaboratory phase of test method development and provided statistically valid results. Information for the suggested analytical wavelengths was collected from each laboratory and has been converted to wavelengths as annotated in the National Institute of Standards and Technology (NIST) Atomic Spectra Database.[4] In this database, wavelengths of less than 200 nm were measured in vacuum and wavelengths greater than or equal to 200 nm were measured in air. Software tables for individual instruments may list wavelengths somewhat differently, as instrument optical path atmospheric conditions may vary.

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

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

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

TABLE 1 Suggested Wavelengths/Interferences

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

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

7. Apparatus

7.1 *Inductively Coupled Plasma Atomic Emission Spectrometers*—Used to perform analysis by this test method may conform to the specifications given in Practice E1479.

Suitability of a specific instrument for testing to this test method will be established using the performance criteria described in 12.1. The sample introduction system shall be capable of handling solutions containing up to 5 % HF.

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

8. Reagents and Materials

8.1 *Reagents:*

8.1.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

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

8.2 *Calibration Solutions:*

8.2.1 In this test method, calibration is based on laboratory-prepared, alloy matrix-matched calibration solutions. Alloy matrix-matched calibration solutions are solutions that contain the approximate amounts of the major alloying elements nickel, chromium, cobalt, molybdenum, and iron found in typical sample solutions. They are intended to model the physical behavior of sample solutions in the plasma. The matrix solutions are prepared with starting materials of known purity and are then spiked with aliquots of single element certified reference material (CRM) solutions that contain the analytes to be quantified. The CRMs shall be compliant with ISO Guide 31 and ISO Guide 34. It may be possible to analyze different alloys using common matrix-matched calibration solutions provided method validation studies demonstrate acceptable data.

8.2.2 Steps 8.2.3 and following describe the preparation of alloy matrix-matched calibration solutions for analysis of sample solutions that contain 1 g alloy/100 mL final dilution. It is acceptable to vary the sample weight and final volume as long as the user's method demonstrates adequate sensitivity and precision (see 12.1).

8.2.3 Calculate the nominal amounts of the alloying metals nickel, chromium, cobalt, molybdenum, and iron in 1 g of the

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alloy to be analyzed. Use a source of each metal that contains a known, low concentration of each analyte to be determined.

8.2.4 Calculate the amount of analyte contained in each matrix metal. This quantity of analyte will be present in the calibration solutions. Total the amount of analyte from these sources and adjust the stated concentration of each calibration solution accordingly.

NOTE 1—Powdered metals have been found acceptable for preparing matrix solutions. Select powdered metals that do not exhibit excessive surface oxidation. However, do not use powdered metals to make analyte additions as oxidation can lead to significant error in the amount of metal added.

8.2.5 Determine the number and concentration of the 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, their lowest concentration near the lowest expected sample concentration, a concentration near the mid range of the expected sample concentrations, and a blank. Regardless, a minimum of three solutions must be used for calibration.

8.2.6 Prepare the alloy matrix-matched solutions as follows:

8.2.6.1 Weigh the amounts of the pure metals calculated in 8.2.3 into a polytetrafluoroethylene beaker. Use one beaker for each calibration solution to be made.

8.2.6.2 Dissolve the pure metals in 20 mL of acid mixture per gram of sample. Select acid mixtures that will dissolve the metals used in the calibration solutions and the alloys to be analyzed using this test method.

8.2.6.3 A mixture of $HCl + HNO_3$ (9 + 1) or $HCl + H_2O + HNO_3$ (3 + 2 + 1) will dissolve many types of nickel alloys. For alloys containing > 5 % molybdenum or > 20 % chromium, or both, it has been found that concentrated HCl with the addition of concentrated HNO_3 dropwise may be necessary to avoid passivation. (**Warning**—If powdered metals are used, add the acid cautiously as powdered metals tend to be very reactive.)

8.2.6.4 It may be necessary to dissolve the pure chromium separately in HCl (1 + 1), as unalloyed chromium does not dissolve readily in the noted acid mixtures. Heat the beakers gently until the metals dissolve. Remove the beakers from the heat, add ten drops of 49 % HF, and swirl gently.

8.2.6.5 If the solutions are being used for the determination of niobium, tantalum, titanium, tungsten, or zirconium, or combinations thereof, then increase the amount of 49 % HF to 2 mL.

8.2.6.6 Transfer the solutions into 100 mL plastic volumetric flasks. Polypropylene or polymethylpentene flasks are acceptable for this purpose.

8.2.6.7 If an internal standard is used, pipet the predetermined amount into each volumetric flask.

8.2.6.8 Proceed to 8.2.8.

8.2.7 As an alternative to using high purity metals for preparing the alloy matrix solution, single element CRM solutions may be used according to the following steps:

8.2.7.1 Calculate the nominal amounts of nickel, chromium, cobalt, molybdenum, and iron in 1 g of the alloy to be analyzed.

8.2.7.2 Transfer appropriate quantities of the single element CRMs into the appropriate number (see 8.2.5) of polytetrafluoroethylene beakers.

8.2.7.3 Heat the beakers gently to the boiling point. Remove the beakers from the heat, add ten drops of 49 % HF, and swirl gently.

8.2.7.4 If the solutions are being used for determination of niobium, tantalum, titanium, tungsten, or zirconium, or combinations thereof, then increase the amount of 49 % HF to 2 mL.

8.2.7.5 If an internal standard is used, pipet the predetermined amount into each volumetric flask.

8.2.7.6 Transfer the solutions into 100 mL plastic volumetric flasks. Polypropylene or polymethylpentene flasks have been found acceptable for this purpose.

8.2.7.7 The CRM solutions used to prepare the matrix solutions may contain analyte elements in significant concentrations. Calculate the amount of analyte contained in each single element CRM addition. Total the amount of analyte from these sources and adjust the stated concentration of each calibration solution accordingly. Proceed to 8.2.8.

8.2.8 Pipet the needed amount of single element CRM solutions into the volumetric flasks, making sure to leave one analyte-free for use as a blank. Adjust the acidity to approximate the acidity of the sample solutions as prepared in 13.1. Typically, if these solutions are to match samples prepared using 1 g of alloy diluted to 100 mL, the quantity of acids used in 8.2.6.2 will be sufficient to hold all analytes in solution. If further dilution is necessary, it may be necessary to adjust the acidity of the calibration and sample solutions to assure solution stability. Dilute the flasks to volume and mix well.

8.3 *Other Materials:*

8.3.1 *Argon*—The purity of the argon shall meet or exceed the specifications of the instrument manufacturer.

8.3.2 *Purge Gases*—The purity of the purge gases shall meet or exceed the specifications of the instrument manufacturer.

8.3.3 *Control Materials:*

8.3.3.1 A laboratory may choose to procure 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 homogenous and well blended. Users of this test method are strongly discouraged from using certified reference materials as routine control materials.

8.3.3.2 A laboratory may find it difficult to procure or have manufactured the materials described in 8.3.3.1 for all of the necessary analytes or alloys. If this is the case, then it is acceptable to prepare equivalent reference material solutions using the procedure described in 8.2 to use as control solutions.

9. Hazards

9.1 This test method involves the use of concentrated HF. Read and follow label precautions, material safety data sheet (MSDS) information, and Practices E50 for HF handling precautions, as well. For precautions to be observed in the use of certain other reagents in this test method, refer to Practices 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 size to allow the weighing of a nominal 1-g sample for dissolution and analysis.

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 and Standardization

12.1 Prior to calibration, 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 12.1.1 and 12.1.2.

12.1.1 *Sensitivity*—Sensitivity shall be evaluated by establishing two-point calibrations for each element being determined using the blank and a high calibration solution prepared as described in 8.2. After thorough rinsing, the blank solution is analyzed ten times. Calculate three times the standard deviation of this determination as an approximation of the detection limit. Calculate ten times the standard deviation to approximate the limit of quantification. If the instrument/parameter selection of the user does not produce an estimated detection limit 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 two-point calibration generated in 12.1.1, analyze the high calibration solution ten times using the instrument/parameters selected by the method user. Calculate the % Relative Standard Deviation (% RSD) as follows:

$$\% \, \mathrm{RSD} = \frac{100 \, s}{\bar{C}} \quad (1)$$

where:

s = estimated standard deviation, and
$\bar{C}$ = average of the ten results for the measured concentration.

12.1.2.1 The calculated % RSD should be approximately 1 %. However, as concentrations decrease or as intensities approach detector saturation, % RSD may tend to increase, while not necessarily affecting the quality of the reported result. During the interlaboratory study % RSD values were typically approximately 1 %, although some values approached 5 %. The user of this test method must decide if precision is adequate for meeting data quality objectives. Practice E1479 provides limited guidance as to the parameters that may have an effect on instrument precision. 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:*

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 inductively coupled plasma atomic emission spectrometry (ICP-AES) 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 two replicates is specified.

12.2.4 Calibrate the instrument using the calibration solutions. Calibration curves for ICP-AES 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 in accordance with Section 15 may help the lab in selecting an appropriate calibration algorithm.

12.2.5 The user of this test method must verify the quality of the calibration fit. Typical ICP-AES 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. If the user elects to use a linear equation with a calculated intercept then the correlation coefficient (r) is calculated by the following equation:

$$r_{xy} = \frac{n \sum_{i=1}^{n} X_i Y_i - \sum_{i=1}^{n} X_i \sum_{i=1}^{n} Y_i}{\sqrt{n \sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i \right)^2} \sqrt{n \sum_{i=1}^{n} Y_i^2 - \left(\sum_{i=1}^{n} Y_i \right)^2}} \quad (2)$$

where:

X = concentration,
Y = intensity,
n = number of standards including the blank, and
i = 1, 2,…n.

12.2.5.1 The user is cautioned that when using this test method it is possible to have a correlation coefficient of 1.0 and still have significant error in the calibration curve. In this case, one or more points may yield calculated concentration values that disagree with the known values by a margin greater than the uncertainty goal set by the user. The user is advised to inspect all points and evaluate the potential for an unacceptable bias in a certain type of alloy. The user of this test 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.

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 a polytetrafluoroethylene beaker.

13.2 Add 20 mL of the same acid mixture used to prepare the calibration solutions (8.2.6) and cover with a polytetrafluoroethylene watchglass.

13.3 Heat the beaker gently until the sample is dissolved.

13.4 Remove the beaker from the heat, add ten drops of 49 % HF, and swirl gently.

13.5 If the solutions are being used for determination of niobium, tantalum, titanium, tungsten, or zirconium, or combinations thereof, then increase the amount of 49 % HF to 2 mL.

NOTE 2—For alloys containing tungsten in excess of the quantification range (> 0.11 %) or significant quantities of molybdenum, residues of tungstic acid, or molybdic acid, or combinations thereof, may be present in the sample solution. These solutions must not be used for tungsten or molybdenum determination.

13.6 If HCl (1 + 1) was used to dissolve the chromium used in the calibration solutions, it will be necessary to adjust the acidity of the sample solution accordingly. It is recommended that this acid be added after initial sample digestion.

13.7 Cool the solution and filter using a medium porosity filter paper into a plastic volumetric flask. The volume of the sample solution flask must be consistent with the final dilution volume of the calibration solutions.

13.8 Add an internal standard if used in the calibration solutions,

13.9 Make any other necessary acid volume adjustments so that the acidity of the samples matches the acidity of the calibration solutions, dilute to volume and mix well.

NOTE 3—Other potential sample preparation issues that should be considered are given as follows. If sample preparation methods other than those specified are used, a validation study as specified in Section 15 should be used to 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.

(2) Caution should be used when 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.

13.10 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. 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 Prepare a control chart for each control sample. Refer to Practice E1329 for guidance. Users of this test method are strongly discouraged from using certified reference materials as routine control materials.

14.2 Most ICP-AES 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 or reference materials (RMs), or both, using this test 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 test 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 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-AES 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 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 concentrations contained in the sample. For example, if the sample is prepared as 1 g of sample diluted to a final volume of 100 mL solution, an analyte volume fraction of 1.00 mg analyte/L of solution corresponds to a mass fraction of 0.010 % analyte in the sample.

17. Report

17.1 Results shall be reported in accordance with the guidance provided in 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