



Designation: E2465 – 23

Standard Test Method for Analysis of Ni-Base Alloys by Wavelength Dispersive X-Ray Fluorescence Spectrometry¹

This standard is issued under the fixed designation E2465; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the analysis of nickel and cobalt based alloys by wavelength dispersive X-ray fluorescence spectrometry for determination of the following elements:

Element	Composition Range
Aluminum	0.0X to X.XX
Chromium	0.XX to XX.XX
Copper	0.0X to XX.XX
Cobalt	0.XX to XX.XX
Hafnium	0.0X to 0.XX
Iron	0.XX to XX.XX
Manganese	0.XX to X.XX
Molybdenum	0.0X to XX.XX
Nickel	XX.XX to XX.XX
Niobium	0.XX to X.XX
Phosphorus	0.00X to 0.0XX
Silicon	0.0X to 0.XX
Tantalum	0.00X to X.XX
Titanium	0.XX to X.XX
Tungsten	0.XX to X.XX
Vanadium	0.00X to 0.XX

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

1.3 This method has been interlaboratory tested for the elements and quantification ranges specified in 1.1. The ranges in 1.1 indicate intervals within which results have been demonstrated to be quantitative by the interlaboratory study. It may be possible to extend this method to other elements or different composition ranges provided that a method validation study as described in Guide E2857 is performed and that the results of this study show that the method extension is meeting laboratory data quality objectives.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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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1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

- E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
- E1172 Practice for Describing and Specifying a Wavelength Dispersive X-Ray Spectrometer
- E1361 Guide for Correction of Interelement Effects in X-Ray Spectrometric Analysis
- E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
- E1621 Guide for Elemental Analysis by Wavelength Dispersive X-Ray Fluorescence Spectrometry
- 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 Other Documents:

- ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories

2.3 U.S. Government Standards:³

- 10 CFR Part 19 Notices, Instructions and Reports to Workers: Inspection and Investigations
- 10 CFR Part 20 Standards for Protection Against Radiation

3. Terminology

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

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 the U.S. Nuclear Regulatory Commission, Public Document Room, One White Flint North, 11555 Rockville Pike, Rockville, MD 20852-2738, http://www.nrc.gov.

4. Summary of Test Method

4.1 Specimens and calibration reference materials are finished to a clean, uniform surface. These are irradiated by highenergy X-ray photons in a spectrometer designed for this function. Secondary X-rays are fluoresced from the materials. Using analyzing crystals, this radiation is diffracted and directed toward a detector that measures the count rates at specified wavelengths. The output(s) of the detector(s) is integrated or counted for a fixed time or until the counts reach a certain fixed number. Either a fixed-channel (simultaneous) spectrometer or a goniometer (sequential) spectrometer, or an instrument combining both one or more fixed-channels and one or more goniometers shall be used.

4.2 Calibrations for each element to be determined are prepared using reference material measurement responses and assigned mass fractions. Mass fractions of the elements in the specimens are determined by relating the measured responses for the specimens to the calibrations.

5. Significance and Use

5.1 This procedure is suitable for manufacturing control and for verifying that the product meets specifications. It provides rapid, multi-element determinations with sufficient accuracy to assure product quality. The analytical performance data included may be used as a benchmark to determine if similar X-ray spectrometers provide equivalent precision and accuracy, or if the performance of a particular spectrometer has changed.

6. Interferences

6.1 Line overlaps, interelement, and matrix effects or some combination of these exist for some of the scope elements. **Table 1** lists some of the common line overlaps encountered in nickel and cobalt alloys. Modern X-ray spectrometers provide software for generation of mathematical corrections to model the effects of line overlaps, interelement, and matrix interferences. The user of this method may choose to use these mathematical corrections for analysis. Guide **E1621** provides an extensive overview of mathematical interference correction methods which may be available in the spectrometer software.

7. Apparatus

7.1 Specimen Preparation Equipment:

7.1.1 *Surface Grinder*, with abrasive belts or disks capable of providing a flat, uniform surface on the reference materials and test specimens. Aluminum oxide, Silicon carbide, and Zirconium oxide belts and discs with a grit size user between 60 and 180 have been found suitable.

7.1.1.1 The laboratory should consider the potential for surface contamination of the specimens by any media chosen for grinding. For example, the use of an aluminum oxide belt or disk may significantly contaminate the surface of “soft” alloys with aluminum. A surface preparation precision study could help evaluate the media chosen.

7.1.2 *Lathe or milling machine*, as an alternative to abrasive surfacing of test specimens. A lathe or milling machine may be used to produce a uniform surface. Carbide type inserts have been found acceptable tooling for this purpose.

7.2 *Wavelength Dispersive X-ray Spectrometer*, designed for X-ray fluorescence analysis. Refer to Practice **E1172** for a detailed discussion on the spectrometer components necessary for analysis.

8. Reagents and Materials

8.1 *Detector Gases*—Only gas-flow proportional counters require a detector gas. Use the gas and purity of gas specified by the instrument manufacturer. Typical gases specified include P-10 or P-5. P-10 consists of a mixture of 90 % argon and 10 % methane, and P-5 consists of a mixture of 95 % argon and 5 % methane. Other gases may be specified as well.

9. Reference Materials

9.1 *Certified Reference Materials*—These are produced and sold by national metrology institutes, international research institutes, and commercial sources. It is preferred that calibrations be characterized using certified reference materials.

9.2 *Reference Materials*—It is recognized that certified reference materials may not be available to fully cover the calibration ranges required for analysis of the variety of nickel and cobalt alloy systems produced. For this reason, it is acceptable to augment calibrations with non-certified reference materials such as in-house reference materials. Reference materials developed using the guidance of Guide **E2972** may be suitable for this purpose.

10. Hazards

10.1 All governing federal, state, and local regulations shall be observed during installation and operation of X-ray fluorescence spectrometers in the United States. The user should follow the guidelines for safe operation given in equipment manufacturer operating manuals. U.S. Nuclear Regulatory standards for ionizing radiation as found in the Code of Federal Regulations, 10 CFR Part 19 and 10 CFR Part 20 provide recommendations on the safe use of X-ray producing equipment.

10.2 Exposure to excessive quantities of high energy radiation such as those produced by X-ray spectrometers is injurious to health. The operator should take appropriate actions to avoid exposing any part of their body, not only to primary X-rays, but also to secondary or scattered radiation that might be present. The X-ray spectrometer should be operated in accordance with the regulations governing the use of ionizing radiation. Manufacturers of X-ray fluorescence spectrometers generally build appropriate shielding/safety interlocks into X-ray equipment during manufacturing that minimize the risk of excessive radiation exposure to operators. Operators should not attempt to bypass or defeat these safety devices. Only authorized personnel should service X-ray spectrometers.

10.3 *Monitoring Devices*, either film badges or dosimeters may be worn by all operating and maintenance personnel. Safety regulations shall conform to applicable local, state, and federal regulations.

11. Preparation of Reference Materials and Test Specimens

11.1 The user must choose a measurement area or diameter from the options built into the spectrometer. All test specimens and reference materials must have a flat surface of greater diameter than the chosen viewed area.

11.2 Prepare the reference materials and test specimens to provide a clean, flat uniform surface to be exposed to the X-ray beam. One surface of a reference material may be designated by the producer as the certified surface. The same preparation medium shall be used for all reference materials and test specimens.

11.3 Refinish the surface of the reference materials and test specimens as needed to eliminate oxidation.

12. Preparation of Apparatus

12.1 Prepare and operate the spectrometer in accordance with the instrument manufacturer's guidance.

NOTE 1—It is not within the scope of this test method to prescribe minute details relative to the preparation of the apparatus. For detailed operating protocols pertaining to a specific spectrometer, refer to the manufacturer's operating manual and published application notes.

12.1.1 *Start-up*—Turn on the power supply and electronic circuits and allow sufficient time for instrument stabilization prior to taking measurements.

12.2 Measurement Conditions:

12.2.1 *Tube Power Supply*—The power supply conditions (kV/mA) should be optimized for the elements being determined.

12.2.1.1 In general, excitation of lighter mass elements is favored by higher current, lower voltage settings and excitation of heavier mass elements is favored by lower current, higher voltage settings. It is desirable, but not mandatory, that the power be held constant for all elements determined via a single program.

12.2.1.2 Once established the optimized current and voltage settings shall be used for generation of calibration curves and for all subsequent specimen measurements.

12.2.2 *Detection System*—The detection systems will consist of masks, collimators, diffraction crystals, and detectors. The crystals, X-ray lines, and detectors specified in **Table 1** have been found to provide acceptable performance for analysis of nickel and cobalt alloys. Set up the instrument to analyze using the available detection systems. Manufacturers are now offering synthetic multi-layer structures which may provide equivalent performance to natural crystals. Alternatives to the detection system configurations described in **Table 1** may be used if performance is acceptable.

TABLE 1 Suggested Detection Parameters

Element	Line Designation ^A	Wavelength (nm) ^B	Crystal	Detector	Potential Line Overlaps
Aluminum	K α	0.8340	PET, EDDT	Sc,FP	
Chromium	K α	0.2291	L1	SP,Sc,FP	V
Cobalt	K α	0.1790	L1,L2	SP,Sc,FP	
Copper	K α	0.1542	L1,L2	SP,Sc,FP	Ta
Hafnium	L α	0.1570	L1,L2	SP,Sc	
Iron	K α	0.1937	L1,L2	SP,Sc	
Manganese	K α	0.2103	L1	SP,Sc,FP	Cr
Molybdenum	K α	0.0711	L1,L2	Sc	
Nickel	K α	0.1659	L1,L2	SP,Sc,FP	
Niobium	K α	0.0748	L1,L2	Sc	
Phosphorus	K α	0.06158	Ge	FP,SP	Mo
Silicon	K α	0.7126	PET,InSb	FP,SP	
Tantalum	L α	0.1522	L1,L2	SP,Sc	
Titanium	K α	0.2750	L1	SP,Sc,FP	
Tungsten	L α	0.1476	L1,L2	SP,Sc	
Vanadium	K α	0.2505	L1	SP,Sc,FP	Ti
Zirconium	K α	0.0787	L1,L2	Sc	

L1 = LiF200
L2 = LiF220
SP = Sealed Proportional
Sc = Scintillation
FP = Flow Proportional

^A Line designations listed in this method are based on the Siegbahn system, which has been superseded by the IUPAC Nomenclature System for X-Ray Spectrometry, Jenkins, R., Manne, R., Robin, R., and Senemaud, C., *Pure & Appl. Chem.*, 63(5), 1991, pp. 735-746.

^B Wavelengths listed in this method are taken from *X-ray and Absorption Wavelengths and Two-Theta Tables*, 2nd Edition; E.W. White and G. G. Johnson, Jr; ASTM Data Series DS 37A; American Society of Testing and Materials; May 1970, pp. inserted wavelength table.

12.2.3 *Counting Time*—Collect a sufficient number of counts so that the precision of the analysis will not be significantly affected by the variation in the number of counts collected. A minimum of 10 000 counts is required for 1 % relative standard uncertainty of X-ray counting and 40 000 for 0.5 % relative standard uncertainty. If fixed time measurements are used, the measurement times can be derived from the

measured intensity (counts per second) and the minimum number of required counts (that is, 10 000 or 40 000). Alternatively, measurement times of 10 s for each of the elements are a good starting point.

12.2.4 Additional spectrometer considerations:

12.2.4.1 Measuring conditions must be selected so that the manufacturer-specified, detector count rate maximum is not

exceeded when materials with mass fractions at the calibration maximum are analyzed. Verify this before analyzing calibration reference materials. Some manufacturers may recommend the use of attenuators to reduce radiation reaching the detector. Use them if necessary.

12.2.4.2 Some manufacturers may recommend the use of filters, for example, primary beam filters or finer detector collimators, to reduce tube radiation reaching the detector. Use them if necessary.

12.2.4.3 When a gas-flow proportional counter is used, adjust the flow of the gas in accordance with the equipment manufacturer's recommendations.

12.2.4.4 Simultaneous instruments will have a fixed crystal and detector assembly for each element being detected. A sequential instrument will have a goniometer that allows crystal and detector combinations to be configured by the laboratory. Some instruments may employ a combination of fixed detector(s) and goniometer(s) to achieve required wavelength coverage. These three instrument designs are allowed by this method.

12.2.4.5 Once the measurement conditions are established, the laboratory shall verify the calibration of the 2-theta angle and the pulse height distribution for each element established in the analytical program.

12.2.4.6 Most sequential spectrometer software allows for the correction of effects of background radiation on the analyte peak intensity. Use of background correction is allowed by this method. If used, one or more "background points" are selected in the region of the analyte peak and counted for a software defined period of time. Care must be taken in the selection of these points, as it is desirable that these points represent the background affecting the analyte intensity. Avoid setting background points on obviously structured regions of the background. The software calculates and applies the correction to the analyte raw intensity.

12.2.5 Ancillary Parameters:

12.2.5.1 Most instruments allow the option of spinning the sample cup. Spin the sample if this option is allowed. If the sample is not rotated during measurement, effects resulting from the interaction of the X-rays with specimen crystal phases or grinding striations or both may significantly bias analytical results. Crystal phases can cause diffraction features that may interfere with analyte peaks or background. Differences in orientation of grinding striations can significantly affect measured count rates.

13. Calibration and Drift Correction (Standardization)

13.1 Calibration:

13.1.1 Using the conditions given in Section 12, measure a series of reference materials covering the required mass fraction range for each element to be quantified. A minimum of three reference materials shall be used for each element. A greater number of reference materials will be required if the user chooses to perform mathematical corrections for interelement effects.

13.1.2 Prepare a calibration curve for each element being determined. Refer to Guide E1621. The instrument software will provide the calibration models used to calculate the calibration curve.

13.1.3 Given the complex nature of nickel and cobalt alloys, it is highly recommended that matrix and line overlap corrections be calculated and applied to the calibration data using the instrument software.

13.1.3.1 Instrument software typically provides a mathematical expression describing the quality of the curve fit. Examples of expressions commonly used are the root mean square difference between known and calculated mass fractions, a chi-square parameter, or correlation coefficient. The root mean square calculation could be augmented to yield a weighted residual error. Refer to the instrument manufacturer's operating guide or other references on statistical mathematics for an in-depth discussion of these calculations.

13.1.3.2 If a significant number of calibration materials are available for an element, it may be possible to calculate the matrix corrections empirically.

13.1.3.3 If the number of calibration materials is minimal, it may be preferable to use matrix corrections calculated using an algorithm based on the fundamental parameters of X-ray fluorescence.

13.1.3.4 Both approaches to generation of calibration curve corrections are allowed by this method.

13.1.3.5 For detailed information on correction of interelement effects in X-ray Spectrometric Analysis refer to Guide E1361. Information on correction of spectral line overlap in wavelength dispersive X-ray spectrometry can be found in Guide E1621.

13.2 *Drift Correction (Standardization)*—Generally a drift correction protocol is established to allow the use of the calibration over an extended period. With a properly designed protocol it may be possible to use a calibration curve set for numerous years. If used, establish the instrument drift correction (standardization) protocol at the time of calibration, using the software function designed for this purpose. Some manufacturers software allows the use of a previously established drift control protocol with new developed measurement methods. In this case, it is acceptable to use the established drift correction (standardization) protocol.

13.2.1 For drift control, materials are measured and the responses are used by the software to calculate mathematical adjustments, either to the slope or both to the slope and intercept of a calibration curve. A correction is typically calculated and applied for each element being determined.

13.2.2 To monitor the calibration slope for changes, select materials which contain a significant amount of the elements being monitored. These materials should generate a count rate on the order of the count rate observed for of the higher mass fraction reference materials used for calibration. The analysis response for these materials is used to calculate the calibration slope adjustment. To monitor the calibration intercept, the lab may select materials with low amount of the elements being monitored. The analytical response of these materials will be used to calculate a calibration intercept adjustment. The count rates for the elements in the drift materials must be in the linear range of the detectors. Additional considerations for material selection are as follows.

13.2.2.1 It is not necessary to use reference materials for drift correction (standardization) since drift correction is based solely on raw count rates (not mass fraction content).

13.2.2.2 It is not necessary that drift correction materials be metals. However, the materials must be demonstrably homogeneous if they will be periodically resurfaced.

13.2.2.3 They must be chemically stable. Alloys and glasses have been found to be suitable materials.

13.2.2.4 The materials must be kept clean and periodic re-surfacing may be necessary to remove dirt and oxidation. It has been observed that silicon and phosphorous are common contaminants introduced to drift material surfaces during repeated use.

13.2.3 Establish a frequency at which the drift control materials will be analyzed. The frequency may be related to an SPC practice or when the detector gas or major components have been changed.

13.2.4 Analyze the drift correction materials. Typically, most software will automatically calculate a slope or slope and intercept correction and apply it to the calibration.

13.3 *Method Validation*—Once an analytical program and calibration curve set is developed, it is recommended that the laboratory perform a method validation study to verify that the analytical method is providing results which meet the laboratory’s data quality objectives. Once validated, it is not necessary to continue to validate the method. Refer to Guide E2857 for guidance concerning method validation.

14. Procedure

14.1 *Specimen Loading*—Place the test specimens in the appropriate specimen holding container (holder). If the spectrometer is equipped with an automated loading device, loading and unloading all specimens from the same holder may improve repeatability. The holder must be equipped to keep the specimen from moving inside the holder.

14.2 *Excitation*—Introduce the holder with specimen to spectrometer.

14.3 *Radiation Measurements*—Obtain and record the counting rate measurement for each element. Either fixed count or fixed time modes may be used. Obtain at least the predetermined minimum counts for all specimens.

14.4 *Replicate Measurements*—Make a minimum of one measurement on each test specimen. The performance of an X-ray spectrometer is not improved significantly by making multiple measurements on the same surface of the specimen. Confidence in the accuracy of analysis may improve by making multiple measurements with each analysis made on a freshly prepared surface.

15. Calculation of Results

15.1 Calculation shall be made using the equipment manufacturer’s software. A brief overview of the specimen analyte mass fraction calculation process is as follows.

15.1.1 Specimen raw intensities are collected and stored in the instrument computer’s database.

15.1.2 The intensities are corrected for measurement response drift, if drift correction is used.

15.1.3 The intensities will be used in conjunction with the matrix and line overlap corrections developed during the calibration process and the appropriate calibration model to calculate the analyte mass fraction. Refer to E1361 and E1621 for detailed discussions on correction and calibration curve calculations.

15.2 Rounding of test results obtained using this Test Method shall be performed as directed in Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specifications.

16. Precision and Bias⁴

16.1 *Precision*—An acceptable number of laboratories tested this method in accordance with Practice E1601. Precision data, including *R* (the Reproducibility Index, see E1601) and *R_{rel}* are provided in Tables 2-14.

16.2 *Bias (Accuracy)*—A number of certified reference materials were tested in the development of this test method. The accuracy of this test method has been deemed satisfactory based on the bias data in Tables 15-27. Users are encouraged to use these or similar reference materials to verify that the test method is performing accurately in their laboratories.

17. Keywords

17.1 cobalt alloys; Nickel alloys; spectrometric analysis; X-ray fluorescence

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1060.

TABLE 2 Statistical Information for Manganese

Test Materials	No. of Labs	% Mn (mean)	R	R _{rel} (%)
J	7	1.5862	0.2319	14.6
D	7	0.7693	0.0505	6.6
G	7	0.5000	0.0268	5.4
B	5	0.4473	0.0380	8.5
O	6	0.3039	0.0153	5.0
P	7	0.2660	0.0159	6.0
E	7	0.2181	0.0965	44.3
H	6	0.2096	0.0172	8.2
I	6	0.1693	0.0116	6.9

TABLE 3 Statistical Information for Phosphorus

Test Material	No. of Labs	% P (mean)	R	R _{rel} (%)
D	5	0.01549	0.00386	24.9
P	5	0.01069	0.00626	58.6
A	5	0.00943	0.00737	78.2
B	5	0.00935	0.00412	43.4
C	5	0.00727	0.00829	114.0
H	5	0.00717	0.00404	56.3
O	5	0.00652	0.00387	59.4
L	5	0.00511	0.00635	124.3

TABLE 4 Statistical Information for Silicon

Test Material	No. of Labs	% Si (mean)	R	R _{rel} (%)
D	7	0.5671	0.0432	7.6
J	7	0.5373	0.0498	9.3
I	7	0.4582	0.0684	14.9
H	7	0.3152	0.0545	17.3
P	7	0.2655	0.0302	11.4
E	7	0.2421	0.0497	20.5
B	7	0.2222	0.0247	11.1
O	7	0.2174	0.0442	20.3
A	7	0.1221	0.0406	33.2
C	7	0.1079	0.0183	17.0
G	7	0.1012	0.0146	14.4
N	7	0.0737	0.0383	52.0
F	7	0.0277	0.0096	34.8
K	7	0.0252	0.0465	184.9

TABLE 5 Statistical Information for Chromium

Test Material	No. of Labs	% Cr (mean)	R	R _{rel} (%)
L	7	21.736	0.399	1.84
N	7	21.733	0.413	1.90
M	7	21.663	0.347	1.60
A	7	21.511	0.300	1.40
B	7	21.146	0.483	2.28
G	7	20.687	0.480	2.32
K	7	20.683	0.451	2.18
I	7	20.602	0.521	2.53
D	7	19.946	0.618	3.10
C	7	18.190	0.344	1.89
O	7	16.265	0.377	2.07
E	7	15.546	0.294	1.89
J	7	15.104	0.310	2.05
P	7	14.870	0.377	2.54
H	7	14.851	0.452	3.04
F	7	11.374	0.401	3.53

TABLE 6 Statistical Information for Nickel

Test Material	No. of Labs	% Ni (mean)	R	R _{rel} (%)
H	6	76.584	1.224	1.60
O	6	75.414	1.110	1.47
E	6	74.800	0.590	0.79
J	6	71.714	0.919	1.28
I	6	62.456	0.706	1.13
L	6	61.774	0.300	0.49
N	6	61.673	0.409	0.66
M	6	61.547	0.337	0.55
A	6	59.544	0.375	0.63
K	6	58.395	0.414	0.71
C	6	53.249	0.375	0.70
P	6	47.453	0.352	0.74
G	6	43.480	0.319	0.73
F	6	41.265	0.505	1.22
B	6	39.972	0.504	1.26
D	6	31.339	0.897	2.86

TABLE 7 Statistical Information for Aluminum

Test Material	No. of Labs	% Al (mean)	R	R _{rel} (%)
P	6	1.2674	0.1293	10.2
J	6	1.0349	0.0532	5.1
C	6	0.5603	0.0398	7.1
L	6	0.3491	0.0503	14.4
A	6	0.3088	0.0219	7.1
D	6	0.2764	0.0473	17.1
F	6	0.2347	0.0414	17.6
H	6	0.2292	0.0524	22.9
N	6	0.2173	0.0313	14.4
M	6	0.2164	0.0314	14.5
E	6	0.1861	0.0327	17.6
B	6	0.1844	0.0474	25.7
I	6	0.1838	0.0491	26.7
O	6	0.1655	0.0812	49.1
G	6	0.1546	0.0180	11.6
K	6	0.1235	0.0997	80.8

TABLE 8 Statistical Information for Molybdenum

Test Material	No. of Labs	% Mo (mean)	R	R _{rel} (%)
I	7	9.8143	1.1866	12.1
A	7	9.4735	1.0329	10.9
L	7	9.3083	1.0381	11.2
N	7	9.1070	0.9867	10.8
M	7	8.7519	0.9041	10.3
K	7	7.8481	0.6813	8.7
P	7	6.3950	0.2690	4.2
F	7	5.8338	0.1567	2.7
C	7	3.0612	0.0474	1.5
B	7	3.0482	0.1083	3.6
G	7	3.0126	0.0889	3.0
J	7	0.5423	0.0524	9.7
D	7	0.1957	0.0341	17.4
E	7	0.0524	0.0126	24.0
H	7	0.0469	0.0134	28.5