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Designation: ~~E2465 – 19~~ E2465 – 23

Standard Test Method for Analysis of Ni-Base Alloys by Wavelength Dispersive X-Ray Fluorescence Spectrometry[1]

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

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

Element	Composition Range
~~Manganese~~	~~0.06 % to 1.6 %~~
~~Phosphorus~~	~~0.008 % to 0.015 %~~
~~Silicon~~	~~0.08 % to 0.6 %~~
~~Chromium~~	~~1.6 % to 22 %~~
~~Nickel~~	~~23 % to 77 %~~
~~Aluminum~~	~~0.20 % to 1.3 %~~
~~Molybdenum~~	~~0.03 % to 10 %~~
~~Copper~~	~~0.007 % to 2.5 %~~
~~Titanium~~	~~0.11 % to 3.0 %~~
~~Niobium~~	~~0.55 % to 5.3 %~~
~~Iron~~	~~0.17 % to 46 %~~
~~Tungsten~~	~~0.06 % to 0.50 %~~
~~Cobalt~~	~~0.04 % to 0.35 %~~

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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

~~NOTE 1—Unless exceptions are noted, ranges can be extended by the use of suitable reference materials. Once these element ranges are extended they must be verified by some experimental means. This could include but not limited to Gage Repeatability and Reproducibility studies, Interlaboratory Round Robin studies, or both. Once these studies are completed, they will satisfy the ISO/IEC 17025 requirements for capability.~~

[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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1.2 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.*

1.5 *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

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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 The test specimen is finished to a clean, uniform surface, then irradiated with an X-ray beam of high energy. The secondary X-rays produced are dispersed by means of crystals and the intensities are measured by suitable detectors at selected wavelengths. The outputs of the detectors in voltage pulses are counted. Radiation measurements are made based on the time required to reach a fixed number of counts, or on the total counts obtained for a fixed time (generally expressed in counts or kilocounts per unit time).

4.1 Mass fractions of the elements are determined by relating the measured radiation of unknown specimens to analytical calibrations prepared with suitable reference materials. 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 sequential goniometer (sequential) spectrometer, or an instrument combining both one or more fixed-channels and one or more goniometers shall be used.

[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.

4.2 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 ~~Interelement or matrix effects~~ Line overlaps, interelement, and matrix effects or some combination of these exist for some of the ~~elements listed. Mathematical~~scope elements. Table 1 ~~correction may be used to solve for these effects. Various mathematical correction procedures are commonly utilized (see~~ 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 ~~E1361~~E1621~~). Any of these procedures that achieves analytical accuracy equivalent to that provided by this test method is acceptable.~~ 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 or Sander With Abrasive Belts or Disks, or Lathe,~~ Grinder,* with abrasive belts or disks capable of providing a flat, uniform surface on the reference materials and test specimens. Aluminum ~~oxide and zirconium~~oxide, Silicon carbide, and Zirconium oxide belts and discs with a grit size ~~of~~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 *Excitation Source:*~~

~~7.2.1 *Tube Power Supply,* providing a constant potential or rectified power of sufficient energy to produce secondary radiation of the specimen for the elements specified. The generator may be equipped with a line voltage regulator and current stabilizer.~~

~~7.2.2 *X-ray Tubes,* with targets of various high-purity elements that are capable of continuous operation at required potentials and currents and that will excite the elements to be determined.~~

7.2 *Wavelength Dispersive X-ray Spectrometer,* designed for X-ray fluorescence ~~analysis and equipped with specimen~~analysis. Refer to Practice E1172 ~~holders and a specimen chamber. The chamber shall contain a specimen spinner, and must be equipped for vacuum or helium-flushed operation for the determination of elements of atomic number 20 (calcium) or lower.~~for a detailed discussion on the spectrometer components necessary for analysis.

~~7.3.1 *Analyzing Crystals,* flat or curved crystals with optimized capability for the diffraction of the wavelengths of interest. The use of synthetic multilayer structures can also be found in some state-of-the-art equipment.~~

~~7.3.2 *Collimators or Slits,* for controlling the divergence of the characteristic X-rays. Use in accordance with the equipment manufacturer's recommendations.~~

~~7.3.3 *Detectors,* sealed-gas, gas-flow, and scintillation counters, or equivalent.~~

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~~7.3.4 *Vacuum System,* providing for the determination of elements whose radiation is absorbed by air (for example, silicon, phosphorus, and sulfur). The system shall consist of a vacuum pump, gage, and electrical controls to evacuate the optical path, and maintain a controlled pressure, usually 13 Pa (100 mm Hg) or less, controlled to ±3 Pa (20 mm Hg). A helium-flushed system is an alternative to a vacuum system.~~

~~7.4 *Measuring System,* consisting of electronic circuits capable of amplifying and integrating pulses received from the detectors. For some measurements, a pulse height selector in conjunction with the detectors may be used to remove high order lines and background. The system shall be equipped with an appropriate device.~~

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~~ 90 % argon and 10 % methane, and P-5 consists of a mixture of ~~95 %~~ 95 % argon and ~~5 %~~ 5 % methane. Other gases may be specified as well.

9. Reference Materials

9.1 *Certified Reference ~~Materials~~ Materials—*~~are available from~~ 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.

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~~9.2 *Reference Materials* with matrices similar to that of the test specimens and containing varying amounts of the elements in the scope of this test method may be used provided they have been analyzed using validated standard methods of test. These reference materials shall be homogeneous and free of voids and porosity.~~

9.2 *Reference Materials—*~~The reference materials shall cover the mass fraction ranges of the elements being sought. A minimum of three reference materials shall be used for each element. A greater number of reference materials may be required if the analyst chooses to perform~~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 ~~mathematical corrections for interelement effects (see Guide~~ may be suitable for this purpose.~~E1361)~~.

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 ~~shall be observed at all X-ray emission spectrometer installations in the United States. It is also recommended that operating and maintenance personnel follow the guidelines of safe operating procedures given in similar handbooks on radiation safety.~~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 ~~analyst~~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 ~~instructions.~~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 ~~a description and specific details concerning the operation of a particular~~detailed operating protocols pertaining to a specific spectrometer, refer to the manufacturer's ~~manual.~~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 ~~warm-up~~stabilization prior to taking measurements.

~~12.2 *Tube Power Supply*—The power supply conditions should be set in accordance with the manufacturer's recommendations.~~

~~12.2.1 The voltage and current established as optimum for the X-ray tube power supply in an individual laboratory shall be reproduced for subsequent measurements.~~

~~12.3 *Proportional Counter Gas Flow*—When a gas-flow proportional counter is used, adjust the flow of the P-10 gas in accordance with the equipment manufacturer's instructions. When changing P-10 cylinders, the detectors should be adequately flushed with detector gas before the instrument is used. After changing P-10 cylinders, check the pulse height selector in accordance with the manufacturer's instructions.~~

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12.2 *Measurement ~~Conditions~~—Conditions:* ~~The K-$L_{2,3}$ (Kα) lines for each element are used, except for tungsten. For tungsten, the L_3-M_5 (Lα) line is used. When using a sequential spectrometer, measurement angles shall be calibrated in accordance with the manufacturer's guidelines.~~

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 ~~*Crystals and Detectors*—~~*Detection System*—The ~~following crystals and detectors are suggested~~detection systems will consist of masks, collimators, diffraction crystals, and detectors. The crystals, X-ray lines, and detectors specified in Table 1~~for the elements indicated:~~ have been found to provide acceptable performance for analysis of nickel and cobalt alloys. Set up the instrument to

~~Element~~	~~Crystal~~	~~Detector~~
~~Chromium~~	~~L1,L2~~	~~SP,Sc,FP~~
~~Cobalt~~	~~L1,L2~~	~~SP,Sc,FP~~
~~Copper~~	~~L1,L2~~	~~SP,Sc,FP~~
~~Manganese~~	~~L1,L2~~	~~SP,Sc,FP~~
~~Molybdenum~~	~~L1,L2~~	~~Sc~~
~~Nickel~~	~~L1,L2~~	~~SP,Sc,FP~~

Element	Crystal	Detector
Niobium	L1,L2	Sc
Phosphorus	Ge	FP,SP
Silicon	PET,InSb	FP,SP
Titanium	L1,L2	SP,Sc,FP
Aluminum	PET	Sc,FP
Iron	L1,L2	SP,Sc
Tungsten	L1,L2	SP,Sc
	L1 = LiF200	SP = Sealed Proportional
	L2 = LiF220	Sc = Scintillation
		FP = Flow Proportional

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	Kα	0.8340	PET, EDDT	Sc,FP	
Chromium	Kα	0.2291	L1	SP,Sc,FP	V
Cobalt	Kα	0.1790	L1,L2	SP,Sc,FP	
Copper	Kα	0.1542	L1,L2	SP,Sc,FP	Ta
Hafnium	Lα	0.1570	L1,L2	SP,Sc	
Iron	Kα	0.1937	L1,L2	SP,Sc	
Manganese	Kα	0.2103	L1,	SP,Sc,FP	Cr
Molybdenum	Kα	0.0711	L1,L2	Sc	
Nickel	Kα	0.1659	L1,L2	SP,Sc,FP	
Niobium	Kα	0.0748	L1,L2	Sc	
Phosphorus	Kα	0.06158	Ge	FP,SP	Mo
Silicon	Kα	0.7126	PET,InSb	FP,SP	
Tantalum	Lα	0.1522	L1,L2	SP,Sc	
Titanium	Kα	0.2750	L1,	SP,Sc,FP	
Tungsten	Lα	0.1476	L1,L2	SP,Sc	
Vanadium	Kα	0.2505	L1,	SP,Sc,FP	Ti
Zirconium	Kα	0.0787	L1,L2	Sc	
			L1 = LiF200	SP = Sealed Proportional	
			L2 = LiF220	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.

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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 counting statistics. number of counts collected. A minimum of 10,000 10 000 counts is required for one percent relative precision of the counting statistics and 40,000 for one-half percent relative. 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). 10 000 or 40 000). Alternatively, measurement times of 10 s 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.