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.

Designation: 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 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.*

1.5 *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.

[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.08 on Ni and Co and High Temperature Alloys.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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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.