



Designation: E572 – 21

Standard Test Method for Analysis of Stainless and Alloy Steels by Wavelength Dispersive X-Ray Fluorescence Spectrometry¹

This standard is issued under the fixed designation E572; 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 method² covers the analysis of stainless and alloy steels by wavelength dispersive X-ray Fluorescence Spectrometry for the determination of the following elements:

Element	Range, Mass Fraction %
Chromium	0.5 to 25
Cobalt	0.05 to 0.45
Copper	0.06 to 3.5
Manganese	0.3 to 5.5
Molybdenum	0.02 to 3.5
Nickel	0.6 to 35
Niobium	0.03 to 1.3
Phosphorus	0.01 to 0.03
Silicon	0.1 to 2
Sulfur	0.02 to 0.35
Titanium	0.008 to 0.5
Vanadium	0.02 to 0.25

NOTE 1—Unless exceptions are noted, mass fraction ranges can be extended by using suitable reference materials. Extended ranges must be verified by experimental means. This could include, but not be limited to, Interlaboratory studies, Round Robin exercises, and other validation approaches. See Guide E2857 for additional guidance.

1.2 *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.* Specific precautionary statements are given in Section 10.

1.3 *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.*

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 E01.01 on Iron, Steel, and Ferroalloys.

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² Supporting data for this test method as determined by cooperative testing have been filed at ASTM International Headquarters as RR:E01-1118. Contact ASTM Customer Service at service@astm.org.

2. Referenced Documents

2.1 *ASTM Standards:*³

- 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
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E1361 Guide for Correction of Inter-element Effects in X-Ray Spectrometric Analysis
- E1621 Guide for Elemental Analysis by Wavelength Dispersive X-Ray Fluorescence Spectrometry
- E2857 Guide for Validating Analytical Methods

3. Terminology

3.1 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 and then irradiated with an X-ray beam of high energy. The secondary X-rays produced are dispersed by means of crystals and the count rates 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 per unit time). Mass fractions of the elements are determined by relating the measured radiation of unknown specimens to analytical curves prepared using suitable reference materials. Both simultaneous spectrometers containing a fixed-channel monochromator for each element and sequential

³ 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.

spectrometers using a goniometer monochromator can be used for measurement of the elements.

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.

5.2 It is expected that this standard will be employed by analysts knowledgeable in the field of X-ray fluorescence spectrometry and experienced in the use of the apparatus specified in this test method.

6. Interferences

6.1 Interelement effects or matrix effects exist for some of the elements listed. Mathematical correction may be used to solve for these elements. Various mathematical correction procedures are commonly utilized. See Guides E1361 and E1621. Any of these procedures that achieves analytical accuracy equivalent to that provided by this test method is acceptable.

6.2 *Spectral Interferences*—Some X-ray spectrometers will not completely resolve radiation from several element combinations. Therefore, take care when interpreting count rates when both elements are present. Mathematical calculations must be used to correct for the interferences. See the following for a listing of elements with potential interferences.

Analyte Element	Potential Interference
Sulfur	Molybdenum
Phosphorus	Molybdenum
Cobalt	Iron
Vanadium	Titanium
Chromium	Vanadium
Manganese	Chromium

7. Apparatus

7.1 Specimen Preparation Equipment:

7.1.1 *Surface Grinder or Sander with Abrasive Belts or Disks, or Lathe*, capable of providing a flat, uniform surface on the reference materials and test specimens. Aluminum oxide and zirconium oxide belts and discs with a grit size of between 60 and 180 have been found suitable.

7.2 Excitation Source:

7.2.1 *X-ray Tube Power Supply*, providing a constant potential of sufficient energy to produce secondary radiation from 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.

7.3 *Spectrometer*, designed for X-ray fluorescence analysis and equipped with specimen holders and a specimen chamber. The chamber shall contain a specimen spinner, and must be

equipped for vacuum or helium-flushed operation for measurement of elements of atomic number 20 (calcium) and lower.

7.3.1 *Analyzing Crystals*, flat or curved crystals with optimized capability for the diffraction of the wavelengths of interest. Synthetic multilayer structures can be used in place of crystals.

7.3.2 *Collimators or Slits*, for controlling the divergence of the characteristic X rays.

7.3.3 *Detectors*, sealed and gas-flow proportional types, scintillation counters, or equivalent. Some spectrometers may allow for tandem use of two different detectors to increase sensitivity.

7.3.4 *Vacuum System*, providing for the determination of elements whose radiation is absorbed by air (atomic number 20 [calcium] and lower). The system shall consist of a vacuum pump, gauge, and electrical controls to provide automatic pump down of the optical path, and to maintain a controlled pressure, usually 13 Pa (100 μ m Hg) or less, controlled to $\pm$ 3 Pa ($\pm$ 20 μ m Hg) or better. A helium-flushed system is an alternative to a vacuum system, and it must be demonstrated to provide sufficient stability to achieve the demonstrated repeatability performance of this test method.

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 required to provide more accurate measurements. The system shall be equipped with an appropriate device.

8. Reagents and Materials

8.1 *Detector Gas (P-10)*, consisting of a mixture of 90 % argon and 10 % methane, for use with gas-flow proportional counters only.

9. Reference Materials

9.1 *Certified Reference Materials* are available from commercial and government sources.

9.2 *Reference Materials* with matrices similar to those of the test specimens and containing varying amounts of the elements to be determined may be used provided they have been analyzed as directed in ASTM standard methods or similar procedures established by the certifying body. These reference materials shall be homogeneous and free of voids and porosity.

9.3 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 calibration reference materials may be required if the analyst chooses to perform mathematical corrections for interelement effects. See Guide E1361.

10. Hazards

10.1 U.S. Nuclear Regulatory Commission Standards for ionizing radiation as found in the Code of Federal Regulations 10 CFR Part 19, "Notices, Instructions and Reports to Workers: Inspection and Investigations" and 10 CFR Part 20, "Standards