



Designation: **E572–13 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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	1 to 25
Chromium	0.5 to 25
Cobalt	0.05 to 0.45
Copper	0.06 to 3.5
Manganese	0.3 to 5.5
Molybdenum	0.05 to 3.5
Molybdenum	0.02 to 3.5
Nickel	0.7 to 35
Nickel	0.6 to 35
Niobium	0.06 to 1.3
Niobium	0.03 to 1.3
Phosphorus	0.01 to 0.03
Silicon	0.2 to 2
Silicon	0.1 to 2
Sulfur	0.02 to 0.35
Sulfur	0.013 to 0.5
Titanium	0.008 to 0.5
Titanium	0.008 to 0.5
Vanadium	0.04 to 0.25
Vanadium	0.02 to 0.25

NOTE 1—~~Mass~~ Unless exceptions are noted, mass fraction ranges can be extended upward by demonstration of accurate calibrations 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 Interelement 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 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 ~~standard~~ 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.

<u>Analyte Element</u>	<u>Potential Interference</u>
Sulfur	<u>Molybdenum</u>
Phosphorus	<u>Molybdenum</u>
Cobalt	<u>Iron</u>
Vanadium	<u>Titanium</u>
Chromium	<u>Vanadium</u>
Manganese	<u>Chromium</u>

7. Apparatus

7.1 Specimen Preparation Equipment:

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.

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 ~~or rectified power~~ 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 ~~(for example, silicon, phosphorus, and sulfur)~~ (atomic number 20 [calcium] and lower). The system shall consist of a vacuum pump, ~~gage, gauge,~~ and electrical controls to provide automatic pump down of the optical path, and to maintain a controlled pressure, usually 13 Pa (100 $\mu\text{m Hg}$) or less, controlled to ± 3 Pa (± 20 $\mu\text{m Hg}$) 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 ~~standard test method~~.

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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 ~~in accordance with~~ 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 ~~calibrants~~ 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 for Protection Against Radiation”⁴ shall be observed at all X-ray emission spectrometer installations in the U.S. It is also recommended that operating and maintenance personnel follow the guidelines of safe operating procedures given in similar handbooks on radiation safety.

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~~ directed in regulations governing the use of ionizing radiation. During manufacturing, manufacturers of X-ray fluorescence spectrometers generally build into X-ray equipment appropriate shielding and safety interlocks 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.

11. Preparation of Reference Materials and Test Specimens

11.1 The analyst 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 primary X-ray ~~beam~~beam. One surface of a reference material may be designated by the producer as the certified surface. The same surface preparation medium shall be used for all reference materials and test specimens.

11.3 As needed, refinish the surfaces of the reference materials and test specimens to eliminate oxidation.

12. Preparation of Apparatus

12.1 Prepare and operate the spectrometer ~~in accordance with~~ the manufacturer’s instructions.

NOTE 2—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 spectrometer, refer to the manufacturer’s manual.

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 according to the manufacturer’s recommendations. Choose and set power supply voltage and current settings sufficient to cause fluorescence of the elements in the method scope.~~

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~~ directed in the equipment manufacturer’s instructions. When changing P-10 ~~tanks, cylinders~~, the detectors should be adequately flushed with detector gas before the instrument is used. After changing P-10 ~~tanks, cylinders~~, check pulse height selector and gain settings according to the manufacturer’s instructions.

12.4 *Measurement Conditions*—The $K\alpha$ ($K-L_{2,3}$) lines are used for all elements in this standard. When using a sequential spectrometer, goniometer angle settings shall be calibrated according to the manufacturer’s guidelines.

12.4.1 *Crystals and Detectors*—The following crystals and detector choices are used for the elements indicated:

Element	Crystal	Detector
Chromium	L1, L2	FP, SP, Sc
Cobalt	L1, L2	FP, SP, Sc

⁴ Available from the Nuclear Regulatory Commission, Public Document Room, Mail Stop:OWFN-1 F13, Washington, DC 20555, (800) 397-4209, or via email at PDR.Resource@nrc.gov, or via the website at www.nrc.gov.

Element	Crystal	Detector
Copper	L1, L2	SP, FP, Sc
Manganese	L1, L2	FP, SP, Sc
Molybdenum	L1, L2	Sc, SP
Nickel	L1, L2	SP, FP, Sc
Niobium	L1, L2	Sc, SP
Phosphorus	Ge	FP, SP
Silicon	PET, InSb	FP, SP
Sulfur	Ge	FP, SP
Titanium	L1, L2	FP, SP
Vanadium	L1, L2	FP, SP

L1 = LiF(200), L2 = LiF(220)

FP = Flow Proportional, SP = Sealed Proportional, Sc = Scintillation

12.4.2 *Counting Time*—Collect a sufficient number of counts so that the random nature of X-ray emission and counting does not significantly influence the repeatability of the measurements. A minimum of 10 000 counts is required for a relative counting uncertainty of 1 % at a level of one standard deviation, and 40 000 counts is required for 0.5 % relative uncertainty.

13. Calibration and Standardization Drift Correction (Standardization)

13.1 *Calibration (Preparation of Analytical Curves)*—*Calibration*—Using the conditions established in Section 12, measure a series of reference materials that cover the required mass fraction ranges. Use at least three reference materials for each element. Prepare an analytical curve for each element being determined (refer to Guide E1621). For information on correction of interelement effects in X-ray fluorescence, refer to Guide E1361. Information on correction of spectral line overlaps in wavelength dispersive X-ray spectrometry can be found in Guide E1621.

13.2 *Standardization (Analytical Curve Adjustment)*—*Drift Correction (Standardization)*—Using control reference materials, check the calibration of the X-ray spectrometer at a frequency consistent with the process control practice of the laboratory or when the detector gas or major spectrometer components have been changed. If the calibration check indicates that the spectrometer has drifted, make appropriate adjustments run the drift correction procedure according to the instructions in the manufacturer's manual. manual and retest the control reference material. If the calibration check is not within accepted ranges, then the spectrometer requires evaluation for malfunctions or required maintenance. Refer to Guide E1621 for frequency of verification of standardization.drift correction (standardization).

13.3 *Type Standardization (Optional)*—After calibration and drift correction, type standardization is an analytical technique that may be employed by some laboratories. This is usually performed utilizing the instrument manufacturer's software and recommendations. A type reference material is selected that is similar in composition to the expected composition of the unknown samples. Type standardization should be performed at a frequency interval determined by the laboratory. The type standardization should be verified by analyzing a control sample or the type reference material and applying an approval criterion to the results before analysis of unknown samples.

14. Procedure

14.1 *Specimen Loading*—Place each reference material or test specimen in the appropriate specimen holding container. If the spectrometer is equipped with an automated loading device, repeatability may be improved by loading and unloading all specimens from the same holder. The container shall have a suitable opening to achieve the required precision in an acceptable amount of time. The holder must be equipped to keep the specimen from moving inside the holder.

14.2 *Excitation*—Expose the specimen to primary X-ray radiation ~~in accordance with~~ as directed in Section 12.

14.3 *Radiation Measurements*—Obtain and record the counting rate for each element. Either fixed count or fixed time modes may be used.

14.4 *Spectral Interferences*—~~Some X-ray spectrometers will not completely resolve radiation from several element combinations (for example, molybdenum and sulfur; molybdenum and phosphorus; and iron and cobalt). Therefore, care must be exercised in the interpretation of count rates when both elements are present. Mathematical calculations must be used to correct for the interferences.~~

14.4 *Replicate Measurements*—Make a single 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 on freshly prepared surfaces of the same specimen.

15. Calculation of Results

15.1 Using the count rates for the test specimen and the appropriate analytical curves, calculate the mass fractions of the various elements.

15.1.1 If mathematical calculations must be made to correct the mass fractions for interelement effects, any one of a number of correction procedures may be employed. Refer to the equipment manufacturer’s manual for the applicable procedure for the instrument being used. See Guide [E1361](#).

15.1.2 Mathematical calculations can be used to correct for spectral interferences by including line overlap correction factors in the calibration algorithm. Corrections can be based either on the mass fraction of the interfering element or on the measured count rate of the interfering element.

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 specification.

16. Precision and Bias

16.1 *Precision*—The precision of this test method is based on an interlaboratory study conducted in the 1980s. Each of seven laboratories tested 11 different steel alloy reference materials. Practice [E691](#) was followed for the design of the study and the analysis of the results. The details are given in research report RR:E01-1118.

16.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the “*r*” value for that material; “*r*” is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

16.1.1.1 Repeatability Limits are listed in [Tables 1-12](#) below.

16.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the “*R*” value for that material; “*R*” is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

16.1.2.1 Reproducibility limits are given in [Tables 1-12](#) below.

16.1.3 The ~~above~~ terms (~~repeatability~~ repeatability limit and ~~reproducibility limit~~ reproducibility limit) are used as specified in Practice [E177](#).

16.1.4 Any judgment ~~in accordance with~~ based on statements [16.1.1](#) and [16.1.2](#) would have an approximate 95 % probability of being correct.

TABLE 1 Nickel (%)

Material	Average \bar{X}	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
3	0.2436	0.0038	0.11	0.011	0.31
2	2.118	0.048	0.091	0.14	0.25
11	3.299	0.015	0.061	0.043	0.17
7	8.156	0.027	0.061	0.075	0.17
5	9.237	0.033	0.079	0.094	0.22
8	10.053	0.039	0.067	0.11	0.19
1	10.938	0.031	0.075	0.087	0.21
10	13.247	0.064	0.31	0.18	0.86
6	18.584	0.068	0.58	0.19	1.6
4	21.19	0.11	0.19	0.29	0.53
9	34.11	0.11	0.17	0.31	0.47

TABLE 2 Chromium (%)

Material	Average \bar{X}	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
6	2.067	0.010	0.086	0.029	0.24
2	15.75	0.116	0.34	0.32	0.95
7	17.30	0.054	0.19	0.15	0.53
8	17.72	0.046	0.21	0.13	0.59
1	17.886	0.073	0.21	0.20	0.60
5	18.19	0.060	0.16	0.17	0.44
11	19.05	0.070	0.24	0.20	0.68
9	19.98	0.073	0.27	0.20	0.75
10	21.25	0.085	0.18	0.24	0.51
3	24.12	0.072	0.65	0.20	1.8
4	24.15	0.10	0.42	0.29	1.2

TABLE 3 Manganese (%)

Material	Average \bar{X}	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
3	0.3120	0.0023	0.047	0.0066	0.13
2	0.4242	0.0035	0.033	0.0097	0.093
9	0.4430	0.0039	0.035	0.011	0.098
5	0.7637	0.0049	0.026	0.014	0.074
11	0.9593	0.0050	0.034	0.014	0.095
1	0.9658	0.0077	0.029	0.022	0.080
6	1.4666	0.0053	0.084	0.015	0.24
8	1.6313	0.0051	0.038	0.014	0.11
4	1.6587	0.0079	0.049	0.022	0.14
7	1.735	0.011	0.057	0.031	0.16
10	4.946	0.017	0.16	0.046	0.44

TABLE 4 Copper (%)

Material	Average \bar{X}	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
5	0.0296	0.0012	0.018	0.0033	0.050
3	0.0591	0.0012	0.012	0.0034	0.033
10	0.0809	0.0009	0.016	0.0026	0.046
1	0.1084	0.0012	0.013	0.0033	0.036
11	0.1353	0.0012	0.011	0.0034	0.031
2	0.1615	0.0013	0.0089	0.0035	0.025
8	0.3104	0.0020	0.0052	0.0055	0.014
7	0.3672	0.0020	0.0090	0.0057	0.025
6	0.4419	0.0031	0.021	0.0088	0.057
4	0.5873	0.0032	0.014	0.0089	0.040
9	3.2914	0.0145	0.12	0.0407	0.326

16.2 *Bias*—At the time of the interlaboratory study, a set of certified reference materials was provided for determining the bias of this test method. Bias estimates are represented by the difference, D , in **Tables 13-24** below.

16.3 The precision and bias statements were determined through statistical examination of results from seven laboratories on these 11 reference materials:

Sample 1:	Standard Reference Material (SRM) C1152, U.S. National Institute of Standards and Technology
Sample 2:	SRM 1219, U.S. National Institute of Standards and Technology
Sample 3:	SRM 1267, U.S. National Institute of Standards and Technology
Sample 4:	SRM C1287, U.S. National Institute of Standards and Technology
Sample 5:	Certified Reference Material (CRM) SS467, Jernklororets Sweden
Sample 6:	CRM S20