



Designation: **E572 – 02a (Reapproved 2006)^{ε2} E572 – 12**

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 NOTE—Updated Section 2 Reference Documents in December 2006.~~

~~² NOTE—Made Editorial corrections to 7.3.4 in March 2007.~~

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 %
Element	Range, Mass Fraction%
Chromium	2.0 to 25.0
Cobalt	0.03 to 0.40
Copper	0.05 to 3.50
Manganese	0.3 to 5.0
Molybdenum	0.15 to 3.5
Nickel	0.20 to 35.0
Niobium	0.05 to 1.3
Phosphorus	0.01 to 0.03
Silicon	0.05 to 0.20
Sulfur	0.02 to 0.30
Titanium	0.002 to 0.04
Vanadium	0.03 to 0.25

NOTE 1—Unless exceptions are noted, ~~concentration~~ mass fraction ranges can be extended by the use of suitable reference materials.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 10.

2. Referenced Documents

2.1 ASTM Standards:³

~~E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials~~

~~E305~~~~E177 Practice for Establishing and Controlling Atomic Emission Spectrochemical Analytical Curves~~~~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~~

~~E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method~~

~~E1621 Guide for X-Ray Emission Spectrometric Analysis~~

~~E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition~~

3. Terminology

3.1 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 E01.01 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:E01-1118RR: E-1-1032~~.

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.

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 ~~intensities-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). ~~Concentrations-Mass fractions~~ of the elements are determined by relating the measured radiation of unknown specimens to analytical curves prepared ~~withusing~~ suitable reference materials. ~~A fixed-channel, polychromator system or a sequential, monochromator~~ 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.

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.

7. Apparatus

7.1 Specimen Preparation Equipment:

7.1.1 ~~Surface Grinder or Sander~~ Withwith 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 ~~offrom~~ 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 ~~emission~~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 ~~the determination-measurement~~ of elements of atomic number 20 (calcium) ~~orand~~ lower.

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

7.3.2 Collimators or Slits, for controlling the divergence of the characteristic X rays. ~~Use per the equipment manufacturer's recommendations.~~

7.3.3 Detectors, sealed ~~orand~~ gas-flow proportional ~~type,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). The system shall consist of a vacuum pump, gage, and electrical controls to provide automatic pump down of the optical path, and to maintain a controlled pressure, usually 13 Pa (100 μm Hg) or less, controlled to 63 Pa (620 μm Hg).- 6 3 Pa (6 20 μm Hg) or better. A helium-flushed system is an alternative to a vacuum system.system, and it must be demonstrated to provide sufficient stability to achieve the demonstrated repeatability performance of this standard.

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 national commercial and international government sources.

9.2 *Reference Materials* with matrices similar to ~~that~~those of the test ~~specimens~~specimens and containing varying amounts of the elements to be determined may be used provided they have been analyzed in accordance with 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 ~~concentration~~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 may be required if the analyst chooses to perform mathematical corrections for interelement effects. See Guide ~~E1361, E691~~.

10. Hazards

10.1 ~~OSHA-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~~installations in the U.S. It is also recommended that operating and maintenance personnel follow the guidelines of safe operating procedures given in ~~current handbooks and publications from National Institute of Standards and Technology and the U.S. Government Printing Office~~, or similar handbooks on radiation safety.

10.2 ~~X-ray equipment shall be used only under the guidance and supervision of a responsible, qualified person. 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 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.~~

10.3 *Monitoring Devices*, either film badges or dosimeters⁷ 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 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. ~~b7e8-c9795ba2ecf8/astm-e572-12~~

11.3 ~~Refinish the surface~~As needed, refinish the surfaces 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 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 prior to taking measurements.

12.2 *Tube Power Supply*—The power supply conditions should be set according to the manufacturers 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.

⁴ *Federal Register*, Vol.36, No. 105, May 1971, Section 1910.96 or of latest issue of Subpart G, available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20025; or *National Bureau of Standards Handbook III*, ANSI N43.2-1971.

⁵ Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, <http://www.dodssp.daps.mil>.

⁶ Available from U.S. Department of Health, Education, and Welfare, Rockville, MD 20850.

⁷ The sole source of supply of the apparatus known to the committee at this time is available from Seimens Gammasonics, Inc., 2000 Nuclear Drive, Des Plaines, IL 60018. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee. 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,¹ which you may attend.

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

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

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

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

L1 = LiF(200);
L2 = LiF(220);
L1 = LiF(200), L2 = LiF(220)
SP = Sealed Proportional;
FP = Flow Proportional, SP = Sealed Proportional, Sc = Scintillation
Sc = Scintillation, and
FP = Flow Proportional.

12.4.2 *Counting Time*—Collect a sufficient number of counts so that the precision of the analysis will not be affected by the variation in the counting statistics. random nature of X-ray emission and counting does not significantly influence the repeatability of the measurements. A minimum of 10 000-10 000 counts is required for one percent precision of the counting statistics, 40 000 for one-half percent, 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

13.1 *Calibration (Preparation of Analytical Curves)*—Using the conditions given established in Section 12, measure a series of reference materials that cover the required concentration-mass fraction ranges. Use at least three reference materials for each element. Prepare an analytical curve for each element being determined (refer to Practice Guide E305E1621). For information on correction of interelement effects in X-ray Spectrometric Analysis-fluorescence, refer to Guide E1361. Information on correction of spectral line overlap overlaps in wavelength dispersive X-ray spectrometry can be found in Practice Guide E1621.

13.2 *Standardization (Analytical Curve Adjustment)*—Using a control reference material, materials, check the calibration of the X-ray spectrometer at a frequency consistent with SPC practice-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 according to the instructions in the manufacturer’s manual. Refer to Practice Guide E305E1621 for frequency of verification of standardization.

14. Procedure

14.1 *Specimen Loading*—Place the each reference materials and material or test specimens 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 Section 12.

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

14.5 *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 will may improve by making multiple measurements on freshly prepared surfaces of the same specimen.

15. Calculation of Results

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

15.1.1 If mathematical calculations must be made to correct the concentrations—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 recommended—applicable procedure for the instrument being used. See Guide E1361.

16. Precision and Bias

16.1 *Precision*—An acceptable number of laboratories tested this method in accordance with—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—Precision data, including repeatability (precision within laboratories), was followed for the design of the study and reproducibility (precision between laboratories), the analysis of the R_r results. The details are provided—given in Tables 1-12 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.

TABLE 1 Statistical Information for Chromium

Test Material	No. of Labs	% Chromium	Min SD (σ _M ; E160+)	Reproducibility-SD (σ _R ; E160+)	Reproducibility-Index (R _r ; E160+)	R _{ref} %
-1	6	17.818	0.05258	0.06684	0.306	-1.7
-2	6	15.627	0.04638	0.05578	0.237	-1.5
3	6	23.906	0.06581	0.08487	0.333	-3.9
4	6	24.011	0.09029	0.10822	0.640	-2.7
5	6	18.146	0.04525	0.05693	0.333	-1.8
-6	6	-2.058	0.00819	0.01047	0.254	12.3
7	6	17.240	0.05008	0.06307	0.247	-1.4
8	6	17.664	0.03935	0.04879	0.435	-2.5
9	6	19.952	0.06473	0.07789	0.769	-3.9
10	6	21.191	0.08769	0.10222	0.293	1.4
11	6	19.018	0.05731	0.06784	0.688	-3.6

TABLE 1 Nickel (%)

Material	Average X̄	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 Statistical Information for Copper

Test-Material	No. of Labs	% Copper	Min-SD (s_{M1} , E1604)	Reproducibility-SD (s_{R1} , E1604)	Reproducibility Index (R_1 , E1604)	R_{ref} %
1	6	0.104	0.0012	0.001572	0.013	12.9
2	6	0.160	0.0012	0.001564	0.024	14.8
3	5	0.054	0.0013	0.001682	0.011	24.0
4	6	0.584	0.0034	0.004222	0.035	-6.0
5	5	0.023	0.0013	0.001634	0.015	65.4
6	6	0.441	0.0017	0.002206	0.062	14.0
7	6	0.370	0.0021	0.002723	0.016	-4.3
8	6	0.311	0.0026	0.003222	0.016	-5.4
9	6	3.249	0.0197	0.023712	0.166	-5.1
10	6	0.075	0.0010	0.001252	0.016	-5.1
11	6	0.132	0.0011	0.0011	0.023	17.3

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 Statistical Information for Cobalt

Test-Material	No. of Labs	% Cobalt	Min-SD (s_{M1} , E1604)	Reproducibility-SD (s_{R1} , E1604)	Reproducibility Index (R_1 , E1604)	R_{ref} %
1	7	0.233	0.0027	0.00335	0.028	-12.2
2	6	0.047	0.0013	0.001616	0.049	103.0
3	5	0.029	0.0015	0.001879	0.009	-32.7
4	6	0.292	0.0030	0.003668	0.072	-24.5
5	6	0.024	0.0015	0.001828	0.006	-25.8
6	6	0.018	0.0022	0.002598	0.022	122.6
7	7	0.156	0.0026	0.003287	0.021	-13.2
8	7	0.222	0.0015	0.001982	0.013	-6.0
9	6	0.402	0.0020	0.00254	0.106	26.5
10	6	0.032	0.0017	0.002034	0.019	-60.0
11	5	0.019	0.0012	0.001469	0.007	-37.6

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

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

16.1.4 Any judgment in accordance with statements 16.1.1 and 16.1.2 would have an approximate 95 % probability of being correct.

16.2 *Bias (Accuracy) = Bias*—Bias data calculated in accordance with Practice 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, E1060 are included D, in Tables 13-24 in below Table 13.