Designation: E 572 – 94 (Reapproved 2000)ε1

# Standard Test Method for X-Ray Emission Spectrometric Analysis of Stainless Steel[1]

This standard is issued under the fixed designation E 572; 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.

ε[1] NOTE—Additional research report was added editorially in June 2001.

## 1. Scope

1.1 This test method[2] provides for the X-ray spectrochemical analysis of stainless steels for the determination of the following elements in the ranges indicated:

| Element | Concentration Range, % |
|---|---|
| Chromium | 11.0 to 19.0 |
| Nickel | 0.20 to 13.0 |
| Copper | 0.05 to 3.50 |
| Molybdenum | 0.05 to 3.00 |
| Manganese | 0.40 to 2.00 |
| Columbium (Niobium) | 0.30 to 0.70 |
| Cobalt | 0.05 to 0.50 |

NOTE 1—These concentration ranges can be extended by the use of suitable reference materials. The detection limit for the elements is lower than the listed value. The ranges represent the nominal levels at which this method was tested.

1.2 This test method is applicable to the control analysis of either chill-cast or wrought samples having a diameter of approximately 25 mm.

NOTE 2—Samples of greater or lesser size than those designated may be used with specially designed sample holders.

1.3 Matrix effects exist between the elements listed. To compensate for these effects, a series of calibration curves are required to cover the designated concentration ranges. The composition of the sample must approximate closely one or more of the reference materials used in the calibration curve which is applied. Mathematical corrections may also be used to solve for matrix effects. (Refer to Practice E 1361.) A variety of such systems is commonly used. Any of these are acceptable that will achieve analytical accuracy equivalent to that reported for the method.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

## 2. Referenced Documents

2.1 *ASTM Standards:*

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[3]

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves[3]

E 353 Test Methods for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys[3]

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data[4]

E 1361 Guide for Correction of Interelement Effects in X-ray Spectrometric Analysis[4]

2.2 *Other Documents:*

MNL 7 Manual on Presentation of Data and Control Chart Analysis[5]

## 3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E 135.

## 4. Summary of Test Method

4.1 The sample is finished to a clean uniform surface and then irradiated by an X-ray beam of high energy (short wavelength). The secondary X rays produced are dispersed by means of crystals and the intensities are measured by detectors at selected wavelengths. Data are collected based on the time required to reach a fixed number of counts, on the total counts obtained for a fixed time, or on the integration of voltage for a fixed time. Concentrations of the elements are determined by relating the measured radiation of unknown samples to analytical curves or charts prepared from standard reference materials of known compositions. A fixed channel or polychromator system or a sequential system may be used to provide simultaneous or sequential determinations of elements.

[1] This test method is under the jurisdiction of ASTM Committee E-1 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 Headquarters as RR:E02–1012 and E01–1032.

[3] *Annual Book of ASTM Standards,* Vol 03.05.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[5] *ASTM Manual Series, ASTM, 6th Edition*, 1990.

**TABLE 1 Typical Operating Voltages and Currents**

| Element | Voltage, kV (Current, mA) | | | | |
|---|---|---|---|---|---|
| Chromium | 60 (45) | 60 (24) | 50 (40) | 20 (8) | 40 (8) |
| Nickel | 60 (45) | 60 (32) | 50 (40) | 60 (32) | 40 (16) |
| Copper | 60 (45) | 60 (32) | 50 (40) | 60 (32) | 40 (24) |
| Molybdenum | 60 (45) | 60 (32) | 50 (40) | 60 (32) | 40 (24) |
| Manganese | 60 (45) | 60 (32) | 50 (40) | 60 (32) | 40 (24) |
| Columbium (Niobium) | 60 (45) | 60 (32) | 50 (40) | 60 (32) | 40 (24) |
| Cobalt | 60 (45) | 60 (32) | 50 (40) | 60 (32) | 40 (24) |

## 5. Apparatus

NOTE 3—It is not within the scope of this test method to prescribe all details of equipment to be used. Equipment varies between laboratories.

5.1 *Sample Preparation Equipment*:

5.1.1 *Surface Grinder*, with 60 to 600-grit aluminum oxide belts or disks capable of providing test specimens with a uniform flat finish.

5.2 *Excitation Source*:

5.2.1 *X-Ray Generator*, providing constant potential or rectified power of sufficient energy to produce secondary radiation of the sample for the elements specified. The generator may be equipped with a line voltage regulator and a current stabilizer.

5.2.2 *X-Ray Tubes*, with targets of various high-purity elements, that are capable of continuous operation up to the potentials and currents shown in Table 1.

NOTE 4—X-ray tubes with tungsten, gold, and rhodium targets were used in the testing of this method.

5.3 *Spectrometer*, designed for X-ray emission analysis, using air or vacuum, and equipped with specimen holders and specimen chamber. The chamber should contain a sample spinner.

5.3.1 *Analyzing Crystal*, flat or curved lithium fluoride LiF(200) or LiF(220).

5.3.2 *Collimator*, for limiting the characteristic X-rays to a parallel bundle when flat crystals are used in the instrument. For curved crystal optics, no collimator is necessary.

5.3.3 *Detectors*—Sealed or gas-flow proportional counters or equivalent.

5.3.4 *Vacuum System*, if used, should consist of a vacuum pump,[6] gage, and electrical controls to provide automatic pumpdown of the optical path and to start the analysis at a pressure of 100 μm or less, controllable to ± 20 μm.

5.4 *Measuring System*—An electronic circuit capable of amplifying and integrating pulses received from the detector tube. The system should be equipped with visual and automatic recording devices.

## 6. Reagents

6.1 *Detector Gas* (P-10), consisting of 90 % argon and 10 % methane.

[6] A two-stage mechanical pump meeting the requirements can be purchased from Precision Scientific Co., Chicago, IL 60647, or Sargent-Welch Scientific Co., Skokie, IL 60076.

## 7. Hazards

7.1 Guidelines on ionizing radiation given in *Occupational Health and Safety Standards*[7] shall be observed at all X-ray emission spectrometer installations. It is also recommended that personnel follow the guidelines of safe operating procedures given in the NIST Handbook *X-Ray Protection, HB76,*[8] the booklet *Radiation Safety Recommendations for X-Ray Diffraction and Spectrographic Equipment*,[9] *#MORP* 68-14, by T. M. Moore and D. J. McDonald, 1968, and the U.S. Government Handbook 93, *Safety Standard for Non-Medical X-Ray and Sealed Gamma-Ray Sources,* Part 1, general, or similar handbooks of latest issue.

7.2 X-ray equipment should be used only under the guidance and supervision of a responsible, qualified person.

7.3 Suitable monitoring devices, either film badges or dosimeters, shall be worn by all personnel using the equipment.[10] To meet local, state, and federal radiation standards, periodic radiation surveys of the equipment for leaks and excessive scattered radiation shall be made by a qualified person using an ionization-chamber detector.[11] The personal film badge survey record, the radiation survey records, and a maintenance record shall be available upon request.

7.4 Special precautions for the operator shall be posted.

7.5 X-ray caution signs shall be posted near the X-ray equipment and at all entrances to the radiation area.

7.6 Fail-safe "X-ray On" warning lights shall be used at the X-ray tube.

## 8. Preparation of Reference Materials and Samples

8.1 Grind the samples to provide a flat, clean area over the entire surface to be exposed to the X-ray beam. Adhere rigorously to the preparation technique established.

## 9. Reference Materials

9.1 *Certified Reference Materials* are available from the National Institute of Standards and Technology[12] and other sources.

9.2 *Reference Materials* can be used, provided they are analyzed in accordance with Test Methods E 353.

## 10. Preparation of Apparatus

10.1 *Start-up*—Energize the power supply and electronic circuits for at least ½h prior to taking measurements.

10.2 *Power Supply*—Adjust the voltage of the power supply to produce secondary fluorescence according to the expression:

[7] *Federal Register,* Vol. 36, No. 105, May 29, 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 111,* ANSI N43.2-1971.

[8] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20025.

[9] Available from U.S. Department of Health, Education, and Welfare, Rockville, MD 20850.

[10] Available from Siemens Gammasonics, Inc., 2000 Nuclear Drive, Des Plaines, IL 60018.

[11] A survey meter called Cutie-Pie has been found satisfactory for this purpose and is available from Nuclear Associates, Westbury, Long Island, NY 11590.

[12] Available from National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, MD 20899.