Designation: E 1085 – 95 (Reapproved 2004)

Standard Test Method for X-Ray Emission Spectrometric Analysis of Low-Alloy Steels[1]

This standard is issued under the fixed designation E 1085; 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 wavelength dispersive X-ray spectrometric analysis of low-alloy steels for the following elements:

Element	Concentration Range, %
Nickel	0.04 to 3.0
Chromium	0.04 to 2.5
Manganese	0.04 to 2.5
Silicon	0.06 to 1.5
Molybdenum	0.005 to 1.5
Copper	0.03 to 0.6
Vanadium	0.012 to 0.6
Cobalt	0.03 to 0.2
Sulfur	0.009 to 0.1
Niobium	0.002 to 0.1
Phosphorus	0.010 to 0.08
Calcium	0.001 to 0.007

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:* [2]

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

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E 1361 Guide for Correction of Interelement Effects in X-Ray Spectrometric Analysis

E 1622 Practice for the Correction of Spectral Line Overlap in Wavelength X-Ray Spectrometry

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 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 are measured by suitable detectors at selected wavelengths. 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. Concentrations of the elements are determined by relating the measured radiation of unknown specimens to analytical curves prepared with suitable reference materials. Either a fixed-channel, polychromator system or a sequential, monochromator system may be used to provide simultaneous or sequential determinations of elements.

5. Significance and Use

5.1 This procedure is suitable for manufacturing control and for verifying that the product meets specifications. This test method provides rapid, multielement determinations with sufficient accuracy to ensure product quality and minimize production delays. The analytical performance data 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 X-ray spectrometer has changed.

5.2 Calcium is sometimes added to steel to effect inclusion shape control in order to enhance certain mechanical properties of steel. This test method is useful for determining the residual calcium in the steel after such treatment.

5.2.1 Because calcium occurs primarily in inclusions, the precision of this test method is a function of the distribution of the calcium-bearing inclusions in the steel. The variation of determinations on freshly prepared surfaces will give some indication of the distribution of these inclusions.

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[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry of Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.01 on Iron, Steel, and Ferroalloys.

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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.

6. Interferences

6.1 Interelement or matrix effects may exist for some of the elements in 1.1. Mathematical corrections may be used to solve for these effects. Various mathematical correction procedures are commonly utilized. See Guide E 1361 and Practice E 1622. Any of these procedures is acceptable that will achieve analytical accuracy equivalent to that provided by this test method.

6.2 Because trace amounts of calcium are being determined with this test method, exercise care not to contaminate the specimen. The presence of calcium in the grinding medium will contaminate the specimen to the extent that erratic and incorrect results will be obtained. Therefore, the grinding medium shall be analyzed for calcium and only those materials that are free of calcium shall be used.

7. Apparatus

7.1 *Specimen Preparation Equipment*:

7.1.1 *Surface Grinder or Sander With Abrasive Belts, Disks, or Lathe*, capable of providing a flat, uniform surface on the reference materials and test specimens.

7.1.1.1 When calcium is to be determined, 240-grit, calcium-free silicon carbide belts or disks shall be used.

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 of 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 will excite the elements to be determined.

7.2.2.1 For the determination of calcium only chromium target tubes were tested. Other targets shall be tested prior to use.

7.3 *Spectrometer*, designed for X-ray emission analysis, and equipped with specimen holders and a specimen chamber. The chamber may contain a specimen spinner, and must be equipped for vacuum or helium-flushed operation for the determination of elements of Atomic Number 20 (calcium) or lower.

7.3.1 *Analyzing Crystals*, flat or curved crystals with optimized capability for the diffraction of the wavelengths of interest.

7.3.2 *Collimator*, for limiting the characteristic X rays to a parallel bundle when flat crystals are used in the instrument. For curved crystal optics, a collimator is not necessary.

7.3.3 *Detectors*, sealed or gas-flow proportional-type, scintillation counters, or equivalent.

7.3.4 *Vacuum System*, providing for the determination of elements whose radiation is absorbed by air (for example, calcium, silicon, phosphorus, and sulfur). The system shall consist of a vacuum pump, gage, and electrical controls to provide automatic pumpdown of the optical path and maintain a controlled pressure, usually 13 Pa (100 μm Hg) or less, controlled to ±3 Pa (±20 μm Hg).

7.4 *Measuring System*, consisting of electronic circuits capable of amplifying and integrating pulses received from the detectors. For some measurements, pulse height analyzers may be required to provide more accurate measurements. The system shall be equipped with an appropriate recording 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 the National Institute of Standards and Technology[3] and other international certification agencies.

9.2 *Reference Materials* with matrices similar to that of the test specimen and containing varying amounts of the elements to be determined may be used provided they have been chemically analyzed in accordance with ASTM standard test methods. These reference materials shall be homogeneous, and free of voids or porosity.

9.3 The reference materials shall cover the concentration ranges of the elements being sought. A minimum of three reference materials shall be used for each element.

10. Hazards

10.1 Occupational Health and Safety standards for ionizing radiation[4] shall be observed at all X-ray emission spectrometer installations. It is also recommended that operating and maintenance personnel follow the guidelines of safe operating procedures given in current handbooks and publications from the National Institute of Standards and Technology[5,6] and the U.S. Government Printing Office,[7] 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.

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. 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. The personal film badge survey record, the radiation survey record, and an equipment maintenance record shall be available upon request.

[3] Office of Standard Reference Materials, National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, MD 20899.

[4] ANSI/NBS Handbook 114, *General Safety Standard for Installations Using Non-Medical X-Ray and Sealed Gamma-Ram Sources*, available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[5] NBS Handbook 76, *Medical X-Ray Protection Up to Three Million Volts*, available as NCRP 33 from NCRP Publications, 7910 Woodmont Ave., Suite 1016, Bethesda, MD 20814.

[6] ANSI N43.2-1977/NBS Handbook 111, *Radiation Safety for X-Ray Diffraction and Fluorescence Analysis*, available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[7] Moore, T. M., and McDonald, D. J., *Radiation Safety Recommendations for X-Ray Diffraction and Spectrographic Equipment*, MORP 68-14, 1968, available from National Technical Information Service, Springfield, VA 22161.