Designation: E1085 – 16

Standard Test Method for
Analysis of Low-Alloy Steels by Wavelength Dispersive X-Ray Fluorescence Spectrometry [1]

This standard is issued under the fixed designation E1085; 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 fluorescence analysis of low-alloy steels for the following elements:

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

NOTE 1—Unless exceptions are noted, mass fraction ranges can be extended and additional elements can be included by the use of suitable reference materials and measurement conditions. Deviations from the published scope must be validated by experimental means. See Guide E2857 for information on validation options.

1.2 The values stated in the International System of Units (SI) are to be regarded as standard. The values given in parentheses are mathematical conversions to other units that are provided for information only, because they may used in older software and laboratory procedures.

1.3 *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]

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

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 a primary X-ray beam of high energy. The secondary X-rays produced are dispersed by means of crystals, and the intensities (also called count rates) 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. Mass fractions of the elements are determined by relating the measured intensities from unknown specimens to analytical curves prepared with suitable reference materials. Either a fixed-channel, polychromator system or a sequential, wavelength dispersive monochromator system may be used to provide simultaneous or sequential determinations of elements, respectively.

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[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] 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.

5. Significance and Use

5.1 This test method is suitable for manufacturing control and for verifying that a product meets specifications. This test method provides rapid, multi-element determinations with sufficient accuracy to ensure product quality and to 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 affect inclusion shape 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.

6. Interferences

6.1 Interelement or matrix effects may exist for some elements in 1.1. Mathematical corrections may be used to compensate for these effects. Various mathematical correction procedures are commonly utilized. See Guide E1361 and Guide E1621. Any of these procedures is acceptable that will achieve analytical accuracy equivalent to that provided by this test method.

6.2 Spectroscopic interferences or line overlaps may be observed, if the energy resolution of the measurement conditions is insufficient. Mathematical corrections may be used to calibrate the overlaps and perform corrections. See Guide E1621.

6.3 Because trace amounts of calcium are being determined with this test method, exercise care not to contaminate the specimen. 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 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 *Wavelength Dispersive Spectrometer,* designed for X-ray fluorescence 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.

7.2.1 *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.

7.2.1.1 For the determination of calcium, only chromium target tubes were tested. Other targets may be used provided they produce data that meets the precision and bias in Section .

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

7.2.3 *Collimator,* for limiting the characteristic X-rays to a parallel bundle when flat crystals are used. Curved crystals do not require a collimator.

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

7.2.5 *Vacuum System,* providing for the determination of elements whose radiation is absorbed by air, atomic number 20 (calcium) or lower. 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).

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 metrology institutes, other government organizations, and commercial suppliers.

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 using valid, independent test methods. These reference materials shall be homogeneous, and free of voids or 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. When correction factors for matrix effects and spectroscopic interferences are to be calculated from the empirical data as calibration parameters, at least one additional reference material is needed for each correction parameter. Refer to Guide E1361 and Guide E1621.

NOTE 2—Calculation of correction factors for matrix effects and spectroscopic interferences from X-ray theory can be used to reduce the number of reference materials required for calibration.

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"[3] shall be observed at all X-ray emission spectrometer installations in the U.S. It is also

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[3] Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov

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 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 Prepare the reference materials and test specimens to provide a clean, flat uniform surface to be exposed to the X-ray beam. For abrasive sanding, select a grit size, and use that grit and size exclusively for all 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.

11.2 Refinish the surfaces of the reference materials and test specimens as needed to eliminate surface contamination.

12. Preparation of Apparatus

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

NOTE 3—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 spectrometer and allow sufficient time for instrument stabilization prior to taking measurements.

12.2 *Tube Power Supply*—Adjust the voltage of the power supply to produce optimum conditions.

12.2.1 The voltage and current established as optimum for the 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 the equipment manufacturer's instructions. When changing P-10 tanks, the detectors should be adequately flushed with detector gas and adjusted before the instrument is used.

12.4 *Measurement Conditions*—The K-$L_{2,3}$ (Kα) line is measured for each element. The peak location, when using a scanning spectrometer, should be optimized for each instrument.

12.4.1 *Crystals and Detectors*—The following crystals and detectors used for the elements are listed below. In some spectrometers, multiple detectors may be used simultaneously.

Element	Crystal	Detector[A]
Nickel	LiF(200), LiF(220)	SP, FP, Sc
Chromium	LiF(200), LiF(220)	FP, SP, Sc
Manganese	LiF(200), LiF(220)	FP, SP, Sc
Silicon	PET, InSb	FP
Molybdenum	LiF(200), LiF(220)	Sc, SP
Copper	LiF(200), LiF(220)	SP, FP, Sc
Vanadium	LiF(200), LiF(220)	FP, SP, Sc
Cobalt	LiF(200), LiF(220)	FP, SP, Sc
Sulfur	Ge	FP
Niobium	LiF(200), LiF(220)	Sc, SP
Phosphorus	Ge	FP
Calcium	LiF(200)	FP

[A] SP = Sealed Proportional, Sc = Scintillation, and FP = Flow Proportional.

12.4.2 *Counting Time*—Collect a sufficient number of counts so that the repeatability of the analysis will not be affected by variance in X-ray counting. A minimum of 10 000 counts is required for 1 % relative standard uncertainty and 40 000 counts for 0.5 %.

13. Calibration and Standardization

13.1 *Calibration (Preparation of Analytical Curves)*—Using the conditions given in Section 12, measure a series of reference materials that cover the mass fraction ranges of interest. Prepare an analytical curve for each element being determined. Refer to Guide E1361 and Guide E1621.

13.2 *Standardization (Drift Correction)*—Using a control reference material, check the calibration of the X-ray spectrometer at a frequency consistent with statistical process control practice, or when the detector gas or major components have been changed. If the calibration check indicates that drift has occurred, make appropriate adjustments in accordance with the instructions in the manufacturer's manual.

14. Procedure

14.1 *Specimen Loading*—Orient the reference materials and test specimens in the specimen chamber so that the relationship between the X-ray beam and the grinding striations is the same for all measurements. This is an essential requirement if the spectrometer is not equipped with a specimen spinner, but is not necessary when a spinner is used.

14.2 *Excitation*—Expose the specimen to primary X-rays in accordance with Section 12.

14.3 *Radiation Measurements*—Obtain the count 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 *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 improve by making multiple measurements on freshly prepared surfaces of the same specimen or by analyzing more than one specimen.

15. Calculation of Results

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

TABLE 1 Repeatability and Reproducibility
(For all elements, except Ca, all values have units of %)

Sample	Certificate Value, %	X	s_r	s_R	r	R
Nickel						
1	0.031 ± 0.001	0.031	0.0013	0.0061	0.0037	0.0171
2	0.250 ± 0.004	0.250	0.0024	0.0104	0.0067	0.0292
3	1.30 ± 0.01	1.317	0.0051	0.0421	0.0141	0.1180
4	0.20 ± 0.01	0.203	0.0031	0.0093	0.0087	0.0260
5	0.182 ± 0.005	0.183	0.0026	0.0085	0.0072	0.0239
6	0.202 ± 0.004	0.203	0.0025	0.0089	0.0071	0.0251
7	2.97 ± 0.02	3.006	0.0189	0.1133	0.0530	0.3174
8	0.025	0.032	0.0011	0.0045	0.0032	0.0126
9	0.16	0.157	0.0031	0.0064	0.0087	0.0180
Chromium						
1	1.26 ± 0.01	1.27	0.0044	0.0096	0.0123	0.0270
2	0.25 ± 0.01	0.258	0.0023	0.0108	0.0064	0.0304
3	0.062 ± 0.002	0.067	0.0017	0.0092	0.0048	0.0257
4	2.37 ± 0.03	2.36	0.0159	0.0288	0.0445	0.0806
5	0.157 ± 0.006	0.156	0.0064	0.0113	0.0179	0.0316
6	0.201 ± 0.006	0.201	0.0024	0.0099	0.0066	0.0276
7	0.56 ± 0.008	0.575	0.0038	0.0128	0.0106	0.0360
8	0.015	0.014	0.0010	0.0029	0.0029	0.0081
9	1.96	1.95	0.0319	0.0483	0.0893	0.1353
Manganese						
1	2.11 ±< 0.01	2.113	0.0065	0.0116	0.0183	0.0326
2	0.91 ± 0.03	0.913	0.0053	0.0123	0.0148	0.0346
3	0.322 ± 0.007	0.314	0.0016	0.0025	0.0044	0.0071
4	0.55 ± 0.01	0.564	0.0036	0.0064	0.0102	0.0179
5	0.97 ± 0.006	0.997	0.0041	0.0126	0.0114	0.0353
6	0.75 ± 0.006	0.756	0.0029	0.0102	0.0081	0.0686
7	0.97 ± 0.02	0.908	0.0054	0.0245	0.0151	0.0686
8	0.025	0.023	0.0006	0.0079	0.0016	0.0221
9	0.325	0.319	0.0034	0.0080	0.0095	0.0223
Silicon						
1	0.019 ± 0.003	0.026	0.0036	0.0173	0.0102	0.0484
2	0.235 ± 0.009	0.258	0.0071	0.0544	0.0199	0.1524
3	1.06 ± 0.01	1.14	0.0247	0.1919	0.0690	0.5373
4	0.18 ± 0.01	0.195	0.0059	0.0452	0.0166	0.1265
5	0.175 ± 0.005	0.195	0.0060	0.0439	0.0168	0.1231
6	0.456 ± 0.005	0.503	0.0100	0.0924	0.0281	0.2588
7	0.22 ± 0.01	0.234	0.0063	0.0476	0.0176	0.1334
8	0.14	0.166	0.0076	0.0390	0.0214	0.1091
9	0.75	0.821	0.0203	0.1394	0.0567	0.3902
Molybdenum						
1	0.79 ± 0.01	0.792	0.0021	0.0081	0.0058	0.0225
2	0.090 ± 0.003	0.089	0.0005	0.0006	0.0013	0.0016
3	0.028 ± 0.002	0.026	0.0003	0.0009	0.0009	0.0025
4	0.93 ± 0.01	0.942	0.0025	0.0084	0.0071	0.0235
5	0.087 ± 0.005	0.089	0.0004	0.0008	0.0011	0.0024
6	0.153 ± 0.003	0.149	0.0007	0.0013	0.0019	0.0035
7	0.98 ± 0.01	1.003	0.0023	0.0159	0.0064	0.0444
8	0.005	0.002	0.0002	0.0011	0.0007	0.0032
9	1.35	1.38	0.0091	0.0159	0.0254	0.0444
Copper						
1	0.032 ± 0.002	0.033	0.0008	0.0022	0.0021	0.0062
2	0.109 ± 0.002	0.110	0.0010	0.0021	0.0029	0.0059
3	0.425 ± 0.004	0.428	0.0017	0.0133	0.0048	0.0371
4	0.134 ± 0.005	0.138	0.0013	0.0028	0.0035	0.0078
5	0.060 ± 0.003	0.058	0.0009	0.0023	0.0027	0.0064
6	0.153 ± 0.005	0.152	0.0012	0.0026	0.0033	0.0074
7	0.50 ± 0.01	0.513	0.0025	0.0209	0.0069	0.0585
8	0.035	0.039	0.0010	0.0027	0.0027	0.0074
9	0.10	0.093	0.0019	0.0028	0.0052	0.0078
Vanadium						
1	0.003 ± 0.0006	0.003	0.0005	0.0023	0.0015	0.0065
2	0.072 ± 0.002	0.074	0.0005	0.0043	0.0015	0.0121
3	0.441 ± 0.001	0.442	0.0128	0.0358	0.0358	0.0608
4	0.013 ± 0.000	0.013	0.0006	0.0017	0.0018	0.0048
5	0.002 ± 0.000	0.003	0.0006	0.0024	0.0017	0.0067
6	0.102 ± 0.004	0.102	0.0007	0.0044	0.0021	0.0123
7	0.50 ± 0.01	0.531	0.0040	0.0143	0.0112	0.0401
8		0.002	0.0004	0.0015	0.0012	0.0043
9	0.045	0.038	0.0008	0.0022	0.0023	0.0063
Cobalt						
1	0.179 ± 0.004	0.172	0.0015	0.0277	0.0041	0.0776
2	0.025 ± 0.001	0.027	0.0010	0.0026	0.0028	0.0074
3	0.012 ± 0.002	0.014	0.0009	0.0026	0.0028	0.0074

TABLE 1 *Continued*

Sample	Certificate Value, %	X	s_r	s_R	r	R
4	0.011 ± 0.001	0.010	0.0014	0.0072	0.0039	0.0203
5	0.012 ± 0.003	0.011	0.0007	0.0038	0.0020	0.0107
6	0.042 ± 0.003	0.044	0.0009	0.0037	0.0026	0.0102
7	0.030 ± 0.003	0.027	0.0013	0.0047	0.0035	0.0132
8		0.008	0.0010	0.0057	0.0027	0.0160
9	0.01	0.005	0.0008	0.0060	0.0022	0.0167
Sulfur						
1	0.044 ± 0.002	0.050	0.0012	0.0093	0.0034	0.0259
2	0.022 ± 0.000	0.024	0.0016	0.0068	0.0044	0.0190
3	0.0067 ± 0.0006	0.006	0.0007	0.0016	0.0020	0.0045
4	0.030 ± 0.0015	0.025	0.0009	0.0042	0.0025	0.0118
5	0.088 ± 0.0015	0.085	0.0026	0.0191	0.0073	0.0535
6	0.046 ± 0.0015	0.046	0.0015	0.0117	0.0041	0.0328
7	0.0083 ± 0.0005	0.006	0.0007	0.0016	0.0020	0.0045
8	0.002	0.002	0.0008	0.0016	0.0022	0.0045
9	0.039	0.040	0.0014	0.0073	0.0040	0.0205
Niobium						
1	0.003 ± 0.001	0.002	0.0002	0.0008	0.0005	0.0022
2	0.049 ± 0.002	0.049	0.0004	0.0026	0.0010	0.0072
3	0.102 ± 0.002	0.106	0.0007	0.0072	0.0019	0.0203
4	0.002 ± 0.001	0.001	0.0003	0.0007	0.0008	0.0020
5		0.000	0.0002	0.0002	0.0004	0.0007
6	0.030 ± 0.001	0.028	0.0003	0.0018	0.0008	0.0049
7	0.030 ± 0.001	0.028	0.0003	0.0017	0.0009	0.0048
8	0.000	0.00	0.0001	0.0001	0.0002	0.0003
9		0.021	0.0005	0.0017	0.0015	0.0048
Phosphorus						
1	0.005 ± 0.001	0.008	0.0007	0.0019	0.0019	0.0052
2	0.034 ± 0.001	0.035	0.0007	0.0018	0.0019	0.0049
3	0.074 ± 0.002	0.076	0.0008	0.0033	0.0019	0.0092
4	0.018 ± 0.002	0.019	0.0007	0.0017	0.0021	0.0049
5	0.068 ± 0.004	0.067	0.0008	0.0028	0.0023	0.0082
6	0.023 ± 0.001	0.021	0.0005	0.0015	0.0014	0.0041
7	0.056 ± 0.002	0.060	0.0010	0.0021	0.0028	0.0059
8	0.005	0.004	0.0023	0.0023	0.0065	0.0065
9	0.035	0.033	0.0009	0.0027	0.0025	0.0075
Calcium, μg/g						
1	73	69.5	1.98	2.52	5.54	7.06
2	10	9.6	0.47	1.63	1.31	4.57
3	61	61.8	1.53	1.75	4.29	4.69
4	40	37.9	1.68	1.68	4.69	4.69

15.1.1 If mathematical calculations are required to correct for interelement effects, any one of a number of correction procedures may be employed. Refer to Guide E1361 and the equipment manufacturer's manual for the recommended procedure for the instrument being used.

15.2 Rounding of test results obtained using this test method shall be performed in accordance with 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*—An acceptable number of laboratories tested this test method in accordance with Practice E691. Precision data, including repeatability (precision within laboratories), *r*, and reproducibility (precision between laboratories), *R*, are provided in Table 1.

NOTE 4—Because calcium exists in inclusions that may not be uniformly distributed within a sample, the *r* and *R* values can be expected to be high. To demonstrate the precision of this test method without reflecting the variation in inclusion distribution, three samples were analyzed without resurfacing between analyses. At mass fraction levels of 7 μg/g, 27 μg/g, and 53 μg/g, the *r* values were 0.4 μg/g, 0.5 μg/g, and 0.8 μg/g, respectively. The *R* values were 3.3 μg/g, 3.6 μg/g, and 2.4 μg/g, respectively. While better precision is obtained using this technique, it is not recommended as a general practice, as it may not reflect the general distribution of the calcium in the steel.

16.2 *Bias (Accuracy)*—There is no bias indicated in this test method.

17. Keywords

17.1 low-alloy steel; wavelength dispersive; X-ray fluorescence

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