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An American National Standard

Standard Test Methods, Practices, and Terminology for Chemical Analysis of Steel Products[1]

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This standard has been approved for use by agencies of the Department of Defense.

INTRODUCTION

This standard was prepared to answer the need for a single document that would include all aspects of obtaining and reporting the chemical analysis of steel, stainless steel, and related alloys. Such subjects as definitions of terms and product (check) analysis variations (tolerances) required clarification. Requirements for sampling, meeting specified limits, and treatment of data usually were not clearly established in product specifications.

It is intended that this standard will contain all requirements for the determination of chemical composition of steel, stainless steel, or related alloys so that product specifications will need contain only special modifications and exceptions.

1. Scope

1.1 This standard covers definitions, reference methods, practices, and guides relating to the chemical analysis of steel, stainless steel, and related alloys. It includes both wet chemical and instrumental techniques.

1.2 Directions are provided for handling chemical requirements, product analyses, residual elements, and reference standards, and for the treatment and reporting of chemical analysis data.

1.3 This standard applies only to those product standards which include this standard or parts thereof as a requirement.

1.4 In cases of conflict, the product specification requirements shall take precedence over the requirements of this standard.

1.5 Attention is directed to Practice A 880 when there may be a need for information on criteria for evaluation of testing laboratories.

1.6 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

A 880 Practice for Criteria for Use in Evaluation of Testing Laboratories and Organizations for the Examination and Inspection of Steel, Stainless Steel, and Related Alloys[2]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[3]

E 30 Test Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron[4]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[4]

E 59 Practice for Sampling Steel and Iron for Determination of Chemical Composition[4]

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[4]

E 212 Test Method for Spectrographic Analysis of Carbon and Low-Alloy Steel by the Rod-to-Rod Technique[5]

E 293 Test Method for Spectrographic Determination of Acid-Soluble Aluminum in Low-Alloy Steel By the Solution Technique[5]

E 322 Method for X-Ray Emission Spectrometric Analysis of Low-Alloy Steels and Cast Irons[5]

E 327 Test Method for Optical Emission Spectrometric Analysis of Stainless Type 18-8 Steels by the Point-to-Plane Technique[5]

E 350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron[4]

E 352 Test Methods for Chemical Analysis of Tool Steels and Other Similar Medium- and High-Alloy Steels[4]

E 353 Test Methods for Chemical Analysis of Stainless,

[1] These test methods, practices, and terminology are under the jurisdiction of ASTM Committee A-1 on Steel, Stainless Steel and Related Alloys, and are the direct responsibility of Subcommittee A01.13 on Methods of Mechanical Testing.

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[2] *Annual Book of ASTM Standards*, Vol 01.03.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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

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

Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys[4]
E 354 Test Methods for Chemical Analysis of High-Temperature, Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt Alloys[4]
E 403 Test Method for Optical Emission Spectrometric Analysis of Carbon and Low-Alloy Steel by the Point-to-Plane Technique[5]
E 404 Test Method for Spectrographic Determination of Boron in Carbon and Low-Alloy Steel by the Point-to-Plane Technique[5]
E 415 Test Method for Optical Emission Vacuum Spectrometric Analysis of Carbon and Low-Alloy Steel[5]
E 421 Test Method for Spectrographic Determination of Silicon and Aluminum in High-Purity Iron[5]
E 485 Test Method for Optical Emission Vacuum Spectrometric Analysis of Blast Furnace Iron by the Point-to-Plane Technique[5]
E 548 Guide for General Criteria Used for Evaluating Laboratory Competence[3]
E 572 Test Method for X-Ray Emission Spectrometric Analysis of Stainless Steel[5]
E 663 Practice for Flame Atomic Absorption Analysis[5]
E 743 Guide for Spectrochemical Laboratory Quality Assurance[5]
E 851 Practice for Evaluation of Spectrochemical Laboratories[5]
E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[5]
E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, Oxygen, and Hydrogen in Steel and in Iron, Nickel, and Cobalt Alloys[4]
E 1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry[5]
E 1063 Test Method for X-Ray Emission Spectrometric Determination of Cerium and Lathanum in Carbon and Low-Alloy Steels[5]
E 1086 Test Method for Optical Emission Vacuum Spectrometric Analysis of Stainless Steel by the Point-to-Plane Excitation Technique[5]
E 1087 Practice for Sampling Molten Steel from a Ladle Using An Immersion Sampler to Produce a Specimen for Emission Spectrochemical Analysis[5]
E 1097 Guide for Direct Current Plasma Emission Spectrometry Analysis[5]
E 1184 Practice for Electrothermal (Graphite Furnace) Atomic Absorption Analysis[5]
E 1282 Guide for Specifying the Chemical Compositions and Selecting Sampling Practices and Quantitative Analysis Methods for Metals and Alloys[4]
E 1329 Practice for Verification and the Use of Control Charts in Spectrochemical Analysis[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *Pertaining to Analyses*:

3.1.1.1 *cast or heat (formerly ladle) analysis*—applies to chemical analyses representative of a heat of steel as reported to the purchaser and determined by analyzing a test sample, preferably obtained during the pouring of the steel, for the elements designated in a specification.

3.1.1.2 *product, check or verification analysis*—a chemical analysis of the semifinished or finished product, usually for the purpose of determining conformance to the specification requirements. The range of the specified composition applicable to product analysis is normally greater than that applicable to heat analysis in order to take into account deviations associated with analytical reproducibility (Note 1) and the heterogeneity of the steel.

NOTE 1—All of the chemical analysis procedures referenced in this document include precision statements with reproducibility data with the exception of Test Methods E 30.

3.1.1.3 *product analysis tolerances* (Note 2)—a permissible variation over the maximum limit or under the minimum limit of a specified element and applicable only to product analyses, not cast or heat analyses.

NOTE 2—The term "analysis tolerance" is often misunderstood. It does not apply to cast or heat analyses determined to show conformance to specified chemical limits. It applies only to product analysis and becomes meaningful only when the heat analysis of an element falls close to one of the specified limits. For example, stainless steel UNS 30400 limits for chromium are 18.00 to 20.00 %. A heat that the producer reported as 18.01 % chromium may be found to show 17.80 % chromium by a user performing a product analysis. If the product analysis tolerance for such a chromium level is 0.20 %, the product analysis of 17.80 % chromium would be acceptable. A product analysis of 17.79 % would not be acceptable.

3.1.1.4 *proprietary analytical method*—a non-standard analytical method, not published by ASTM, utilizing reference standards traceable to the National Institute of Standards and Technology (NIST) (when available) or other sources referenced in Section 10.

3.1.1.5 *referee analysis*—performed using ASTM methods listed in 9.1.1 and NIST reference standards or methods and reference standards agreed upon between parties. The selection of a laboratory to perform the referee analysis shall be a matter of agreement between the supplier and the purchaser.

3.1.1.6 *certified reference material*—a specimen of material specially prepared, analyzed, and certified for chemical content under the jurisdiction of a recognized standardizing agency or group, such as the National Institute of Standards and Technology, for use by analytical laboratories as an accurate basis for comparison. Reference samples should bear sufficient resemblance to the material to be analyzed so that no significant differences are required in procedures or corrections (for example, for interferences or inter-element effects).

3.1.1.7 *working reference materials*—reference materials used for routine analytical control and traceable to NIST standards and other recognized standards when appropriate standards are available.

3.1.2 *Pertaining to Elements*:

3.1.2.1 *intentionally added unspecified element*—an element added in controlled amounts at the option of the producer to obtain desirable characteristics.

3.1.2.2 *residual element*—a specified or unspecified element, not intentionally added, originating in raw materials, refractories, or air.

3.1.2.3 *specified element*—an element controlled to a specified range, maximum or minimum, in accordance with the requirements of the product specification.

3.1.2.4 *trace element*—a residual element that may occur in very low concentrations, generally less than 0.01 %.

4. Concerning the Specification of Chemical Composition Requirements

4.1 It is recommended that Guide E 1282 be consulted as a guide for specifying the chemical compositions for steels.

4.2 The recommended practice for specifying chemical composition limits is to limit the number of significant figures for each element so that the number of figures to the right of the decimal point conforms to the following:

Chemical Concentration		Maximum Number of Figures to the Right of the Decimal Point
Up to 0.010 %	—	0.XXXX or may be expressed as ppm
Up to 0.10 %	—	0.XXX
0.10 to 3.00 %	—	0.XX
Over 3.0 %	—	0.X

4.3 For those cases in which the composition range spans either 0.10 % or 3.0 %, the number of figures to the right of the decimal is to be determined by that indicated by the upper limit.

4.4 Technical considerations may dictate the employment of less than the number of figures to the right of the decimal as previously recommended.

NOTE 3—The recommendations should be employed to reduce the number of significant figures, such as from 18.00 % to 18.0 %, but a significant figure should never be added unless there is a technical reason for so doing.

5. Cast or Heat Analysis

5.1 The producer shall perform analyses for those elements specified in the material specification. The results of such analyses shall conform to the requirements specified in the material specification.

5.1.1 For multiple heats, either individual heat or cast analysis or an average heat or cast analysis shall be reported. If significant variations in heat or cast size are involved, a weighted average heat or cast analysis, based on the relative quantity of metal in each heat or cast, shall be reported.

5.1.2 For consumable electrode remelted material, a heat is defined as all the ingots remelted by the same process from a primary heat. The heat analysis shall be obtained from one remelted ingot, or the product of one remelted ingot, from each primary melt. If this heat analysis does not meet the heat analysis requirements of the specification, one sample from the product of each remelted ingot shall be analyzed, and the analyses shall meet the heat analysis requirements.

5.2 If the test samples taken for the heat analysis are lost, inadequate, or not representative of the heat, a product analysis of the semifinished or finished product may be used to establish the heat analysis.

5.2.1 If a product analysis is made to establish the heat analysis, the product analysis shall meet the specified limits for heat analysis and the product analysis tolerances described in Section 6 do not apply.

6. Product Analysis Requirements

6.1 For product analysis, the range of the specified chemical composition is normally greater (designated product analysis tolerances) than that applicable to heat analyses to take into account deviations associated with analytical reproducibility and the heterogeneity of the steel. If several determinations of any element in the heat are made, they may not vary both above and below the specified range.

6.2 Product analysis tolerances may not be used to determine conformance to the specified heat or cast analysis unless permitted by the individual material specification.

6.3 Product analysis tolerances, where available, are given in the individual material specifications or in the general requirement specifications.

7. Unspecified Elements (Note 4)

7.1 Reporting analyses of unspecified elements is permitted.

NOTE 4—All commercial metals contain small amounts of various elements in addition to those which are specified. It is neither practical nor necessary to specify limits for every unspecified element that might be present, despite the fact that the presence of many of these elements is often routinely determined by the producer.

7.2 Analysis limits shall be established for specific elements rather than groups of elements such as “all others,”“ rare earths,” and “balance.”

8. Sampling

8.1 *Cast or Heat Analyses*:

8.1.1 Samples shall be taken, insofar as possible, during the casting of a heat, at a time which, in the producer’s judgment, best represents the composition of the cast.

8.1.2 In case the heat analysis samples or analyses are lost or inadequate, or when it is evident that the sample does not truly represent the heat, representative samples may be taken from the semifinished or finished product, in which case such samples may be analyzed to satisfy the specified requirements. The analysis shall meet the specified limits for heat analysis.

8.2 *Check, Product, or Verification Analyses*—Unless otherwise specified, the latest revision of Practice E 59 shall be used as a guide for sampling.

9. Test Methods

9.1 This section lists some test methods that have been found acceptable for chemical analysis of steels.

9.1.1 The following ASTM wet chemical test methods have been found acceptable as referee test methods and as a base for standardizing instrumental analysis techniques:

Test Methods		General Description
E 30	—	antecedent to Test Methods E 350 through E354
E 350	—	the basic wet chemical procedure for steels
E 352	—	wet chemical procedure for tool steels
E 353	—	wet chemical procedure for stainless steels
E 354	—	wet chemical procedure for high nickel steels
E 1019	—	determination of carbon, sulfur, nitrogen, oxygen, and hydrogen, in steel and in iron, nickel, and cobalt alloys

9.1.2 The following ASTM instrumental test methods, practices, and guides may be employed for chemical analysis of steels or may be useful as a guide in the calibration and standardization of instrumental equipment for routine sampling and analysis of steels:

Standard		General Description
E 50	—	apparatus, reagents, and safety
E 60	—	photometric and spectrophotometric work
E 212	—	spectrographic analysis of steels (rod-to-rod technique)
E 293	—	spectrographic analysis of acid-soluble aluminum
E 322	—	X-ray fluorescence for steels
E 327	—	spectrometric analysis of stainless steels
E 403	—	spectrometric analysis of steels
E 404	—	spectrographic determination of steels for boron (point-to-plane technique)
E 415	—	vacuum spectrometric analysis of steels
E 421	—	spectrographic determination of silicon and aluminum in high-purity iron
E 485	—	optical emission vacuum spectrometric analysis of blast furnace iron by the point-to plane technique
E 572	—	X-ray emission spectrometric analysis of stainless steels
E 663	—	flame atomic absorption
E 882	—	accountability and quality control
E 1019	—	determination of carbon, sulfur, nitrogen, oxygen, and hydrogen in steel and in iron, nickel, and cobalt alloys
E 1024	—	flame atomic absorption
E 1063	—	X-ray emission spectrometric determination of cerium and lathanum in carbon and low-alloy steels
E 1086	—	optical emission vacuum spectrometric analysis of stainless steel by the point-to plane excitation technique
E 1087	—	sampling
E 1097	—	direct current plasma spectroscopy
E 1184	—	graphite furnace atomic absorption
E 1282	—	selecting sampling practices and analysis methods
E 1329	—	verification and use of control charts

9.2 The following are some of the commonly accepted techniques employed for routine chemical analysis of steels. These routine analyses are the basis for the producers' quality control/assurance programs. Proprietary methods are permissible provided the results are equivalent to those obtained from standard methods when applicable.

9.2.1 Analysis of stainless steels using X-ray fluorescence spectroscopy (XRF). See Table 1 for normal elements and ranges for stainless steels.

9.2.2 Analysis of stainless steels using spark emission spectroscopy (OES). See Table 2 for normal elements and ranges for stainless steels.

9.2.3 Analysis of solutions using an atomic absorption spectrophotometer.

9.2.4 Analysis of solutions using an inductively coupled plasma emission spectrometer.

9.2.5 Determination of carbon or sulfur, or both, by combustion (in oxygen) and measurement of CO_2 or SO_2, or both, by thermal conductivity or infrared detectors.

	Element Ranges %			Element Ranges %
C	0.002 — 5.0		S	0.0005 — 0.1

9.2.6 Determination of nitrogen and oxygen by fusion (in a helium atmosphere) and measurement of N_2 by thermal conductivity and oxygen by measurement of CO by infrared or thermal conductivity detectors.

TABLE 1 Normal Elements and Ranges for Stainless Steels Using X-Ray Fluorescence Spectroscopy

Element Ranges %		Element Ranges %	
MN	0.005–15.0	Cu	0.005–4.0
P	0.001–0.15	Cb	0.005–3.0
Si	0.005–5.0	V	0.005–2.0
Cr	0.01–26.0	Ti	0.005–2.5
Ni	0.01–36.0	Co	0.005–4.0
Al	0.002–5.5	Sn	0.002–0.20
Mo	0.005–8.0	W	0.005–3.0

TABLE 2 Normal Elements and Ranges for Stainless Steels Using Spark Emission Spectroscopy

C	0.004–5.0	V	0.005–2.0
S	0.0005–0.1	Ti	0.005–2.5
N_2	0.0020–0.3	Co	0.005–4.0
MN	0.005–15.0	Sn	0.001–0.20
P	0.001–1.5	W	0.005–3.0
Si	0.005–5.0	Pb	0.002–0.05
Cr	0.01–26.0	B	0.0005–0.05
Ni	0.01–36.0	Ca	0.0002–0.01
Al	0.001–5.5	Mg	0.001–0.01
Mo	0.005–8.0	Ce	0.001–0.2
Cu	0.005–4.0	Zr	0.001–0.1
Cb	0.005–3.0	Ta	0.005–0.5

	Element Ranges %		
N_2	0.0005	—	0.3
O_2	0.0008	—	0.02

9.2.7 Analysis of solutions using inductively coupled plasma emission spectroscopy (ICP) or direct plasma emission spectroscopy (DCP). Normal elements and ranges for stainless steels are as follows:

	Element Ranges %		
B	0.0002	—	0.01
Ca	0.0002	—	0.01
Mg	0.0002	—	0.01
Ce	0.001	—	0.2
Zr	0.001	—	0.1
Ta	0.005	—	0.5
La	0.001	—	0.01

9.3 There are additional common techniques often used for chemical analysis of standards for instrument analysis such as: polarographic analysis, ion exchange separations, radioactivation, and mass spectrometry.

10. Reference Materials

10.1 For referee analyses, reference standards of a recognized standardizing agency shall be employed with preference given to NIST standard reference materials when applicable. (NIST does not produce reference standards suitable for all elements or all alloys.[6])

10.1.1 When standard reference materials for certain alloys are not available from NIST, reference materials may be produced by employing ASTM standard procedures and NIST standard reference materials to the extent that such procedures and reference standards are available. Several independent laboratories should be used for certification of these standards and their results statistically reviewed and merged.

10.1.2 Methods not published by ASTM such as a definitive analytical method may be used when the method is validated by analyzing certified reference materials along with the candidate reference material. Examples of definitive analytical methods include gravimetric, coulometry, titrimetric based on normality, and mass spectrometry.

10.2 Working reference materials may be used for routine analytical control.

11. Significant Numbers

11.1 Laboratories shall report each element to the same

[6] Some sources of reference materials are listed in ASTM Data Series Publication No. DS2, issued 1963.

number of significant numbers as used in the pertinent material specifications.

11.2 When a chemical determination yields a greater number of significant numbers than is specified for an element, the result shall be rounded in accordance with Section 12.

12. Rounding Procedure

12.1 To determine conformance with the specification requirements, an observed value or calculated value shall be rounded in accordance with Practice E 29 to the nearest unit in the last right-hand place of values listed in the table of chemical requirements.

12.2 In the special case of rounding the number "5" when no additional numbers other than "0" follow the "5", rounding shall be done in the direction of the specification analysis limits if following Practice E 29 would cause rejection of material.

13. Report

13.1 The chemical analysis of a heat shall be reported only when specified by the user or the material specification.

NOTE 5—Columbium, Cb, is also known as niobium, Nb.

13.2 Certification or identification to an ASTM material standard requires that the specific heat of steel meets the chemical requirements of the ASTM standard.

13.3 Any report that is legally binding on the manufacturer is acceptable as a certified test report.

13.3.1 Notarization of Certificates of Test is neither required nor prohibited.

14. Keywords

14.1 cast analysis; chemical analysis; heat analysis; product analysis; reference materials

APPENDIX

(Nonmandatory Information)

X1. QUALITY ASSURANCE FOR VALIDITY OF ANALYTICAL RESULTS

X1.1 The requirements embodied in Guide E 548 and Practices A 880 and E 851, provide generic requirements for production of valid chemical-analysis results.

X1.2 Additional pertinent standards for improving the competency of chemical analysis laboratories are included in Guides E 743 and E 882.

X1.3 Keys to improving validity of chemical analytical results are as follows:

X1.3.1 Replication of sampling and testing to improve the precision of results;

X1.3.2 Use of reference materials is crucial to accurate results;

X1.3.3 Instrumentation that is appropriate and properly maintained; and

X1.3.4 Personnel who are properly trained, ethical chemists or technicians and who work with properly documented, current standards.