

TEST METHODS AND PRACTICES FOR CHEMICAL ANALYSIS OF STEEL PRODUCTS



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(Identical with ASTM Specification A751-21 except for editorial corrections to an element designation in Tables 1 and 2.)

Test Methods and Practices for Chemical Analysis of Steel Products

INTRODUCTION

These test methods and practices were 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 these test methods and practices 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 These test methods and practices cover definitions, reference methods, practices, and guides relating to the chemical analysis of steel, stainless steel, and related alloys. They include 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 These test methods and practices apply only to those product standards which include these test methods and practices, or parts thereof, as a requirement.

1.4 In cases of conflict, the product specification requirements shall take precedence over the requirements of these test methods and practices.

1.5 Attention is directed to ISO/IEC 17025 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:

- A941 Terminology Relating to Steel, Stainless Steel, Related Alloys, and Ferroalloys
- E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
- E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
- E322 Test Method for Analysis of Low-Alloy Steels and Cast Irons by Wavelength Dispersive X-Ray Fluorescence Spectrometry (Withdrawn 2021)
- E350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron
- E352 Test Methods for Chemical Analysis of Tool Steels and

Other Similar Medium- and High-Alloy Steels
 E353 Test Methods for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys
 E354 Test Methods for Chemical Analysis of High-Temperature, Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt Alloys
 E415 Test Method for Analysis of Carbon and Low-Alloy Steel by Spark Atomic Emission Spectrometry
 E548 Guide for General Criteria Used for Evaluating Laboratory Competence (Withdrawn 2002)
 E572 Test Method for Analysis of Stainless and Alloy Steels by Wavelength Dispersive X-Ray Fluorescence Spectrometry
 E743 Guide for Spectrochemical Laboratory Quality Assurance (Withdrawn 1998)
 E851 Practice for Evaluation of Spectrochemical Laboratories (Withdrawn 1998)
 E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
 E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Inert Gas Fusion Techniques
 E1085 Test Method for Analysis of Low-Alloy Steels by Wavelength Dispersive X-Ray Fluorescence Spectrometry
 E1086 Test Method for Analysis of Austenitic Stainless Steel by Spark Atomic Emission Spectrometry
 E1097 Guide for Determination of Various Elements by Direct Current Plasma Atomic Emission Spectrometry
 E1184 Practice for Determination of Elements by Graphite Furnace Atomic Absorption Spectrometry
 E1282 Guide for Specifying the Chemical Compositions and Selecting Sampling Practices and Quantitative Analysis Methods for Metals, Ores, and Related Materials
 E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis (Withdrawn 2019)
 E1476 Guide for Metals Identification, Grade Verification, and Sorting
 E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition
 2.2 *ISO Standards:*
 ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in these test methods and practices, see Terminology A941.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *product, check, or verification analysis, n*—a chemical analysis of the semifinished or finished product, usually for the purpose of determining conformance to the specification requirements.

3.2.1.1 *Discussion*—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 these test methods include precision statements with reproducibility data, with the exception of Test Methods E50.

3.2.2 *product analysis tolerances, n*—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).

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.

3.2.3 *proprietary analytical method, n*—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.

3.2.4 *referee analysis, n*—performed using ASTM test methods listed in 9.1.1, NIST reference standards or methods, and reference standards agreed upon between parties.

3.2.4.1 *Discussion*—The selection of a laboratory to perform the referee analysis shall be a matter of agreement between the supplier and the purchaser.

3.2.5 *certified reference material, n*—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 NIST, for use by analytical laboratories as an accurate basis for comparison.

3.2.5.1 *Discussion*—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).

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

4. Concerning Specification of Chemical Composition Requirements

4.1 It is recommended that Guide E1282 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 Right of Decimal Point
Up to 0.010 %, incl.	0.XXXX or may be expressed as ppm
Over 0.010 % to 0.10 %, incl	0.XXX
Over 0.10 % to 3.0 %, incl	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.

5.3 Unless otherwise specified, compositions shall be reported in mass fraction percent (wt%).

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 E1806 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
E350	Basic wet chemical procedure for steels.
E352	Wet chemical procedure for tool steels.
E353	Wet chemical procedure for stainless steels.
E354	Wet chemical procedure for high nickel steels.
E1019	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.

Test Methods, Practices, Guides	General Description
E50	Apparatus, reagents, and safety.
E60	Photometric and spectrophotometric work. Spectrographic analysis of steels (rod-to-rod technique). Spectrographic analysis of acid-soluble aluminum.
E322	X-ray fluorescence for steels Spectrometric analysis of stainless steels
E415	Vacuum spectrometric analysis of steels Spectrographic determination of silicon and aluminum in high-purity iron.
E572	X-ray emission spectrometric analysis of stainless steels. Flame atomic absorption.
E882	Accountability and quality control.
E1019	Determination of carbon, sulfur, nitrogen, oxygen, and hydrogen in steel and in iron, nickel, and cobalt alloys.
E1085	X-ray emission spectrometric analysis of low alloy steels.
E1086	Optical emission vacuum spectrometric analysis of stainless steel. By the point-to plane excitation technique.
E1097	Direct current plasma spectroscopy.
E1184	Graphite furnace atomic absorption.
E1282	Selecting sampling practices and analysis methods.
E1329	Verification and use of control charts.
E1806	Sampling.

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 CO₂ or SO₂, or both, by thermal conductivity or infrared detectors.

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 N₂ by thermal conductivity and oxygen by measurement of CO by infrared or thermal conductivity detectors.

Element Ranges %	
N ₂	0.0005–0.3
O ₂	0.0008–0.02

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

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.)

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 Test 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 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 E29 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 E29 would cause rejection of material.

13. Records

13.1 In addition to the test data requested, the test records shall contain the following information as appropriate:

13.1.1 Description of the material tested, for example, heat number, grade of material, product specification, and

13.1.2 Test method(s) or unambiguous description of the nonstandard method(s) used.

14. Keywords

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

APPENDIXES

(Nonmandatory Information)

X1. QUALITY ASSURANCE FOR VALIDITY OF ANALYTICAL RESULTS

X1.1 The requirements embodied in Guide E548, ISO/IEC 17025, and Practice E851 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 E743 and E882.

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.

X2. DISCUSSION OF POSITIVE MATERIAL IDENTIFICATION (PMI)

X2.1 PMI is not a true analysis method comparable to the test methods described in the body of this standard and, therefore, is not to be used for reportable analysis of material chemical composition. This appendix is included for reference purposes only and does not allow PMI to be used for the purpose of analysis where these test methods and practices are referenced in other standards.

X2.2 PMI typically utilizes portable instruments to determine material type for the purpose of identification and sorting.

X2.3 PMI can provide accurate non-destructive identification of many material types.

X2.4 PMI is intended for material identification and for

sorting by material type.

X2.5 PMI is limited as to the elements reported. Lighter elements may not be reported or, if reported, may have a large uncertainty.

X2.6 The applicable ASTM standard for PMI is Guide E1476. Users are directed to Guide E1476 for additional instruction.

X2.7 In addition to Guide E1476, the user should refer to the instructions from the manufacturer of the specific instrument to determine the operation, capabilities, and limitations of that instrument.