



Designation: E2994 – 21

Standard Test Method for Analysis of Titanium and Titanium Alloys by Spark Atomic Emission Spectrometry and Glow Discharge Atomic Emission Spectrometry (Performance-Based Method)¹

This standard is issued under the fixed designation E2994; 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 describes the analysis of titanium and its alloys by spark atomic emission spectrometry (Spark-AES) and glow discharge atomic emission spectrometry (GD-AES). The titanium specimen to be analyzed may be in the form of a disk, casting, foil, sheet, plate, extrusion, or some other wrought form or shape. The elements and ranges covered in the scope by spark-AES of this test method are listed below.

Element	Tested Mass Fraction Range (%)
Aluminum	0.008 to 7.0
Chromium	0.006 to 0.1
Copper	0.014 to 0.1
Iron	0.043 to 0.3
Manganese	0.005 to 0.1
Molybdenum	0.014 to 0.1
Nickel	0.006 to 0.1
Silicon	0.018 to 0.1
Tin	0.02 to 0.1
Vanadium	0.015 to 5.0
Zirconium	0.013 to 0.1

1.1.1 The elements oxygen, nitrogen, carbon, niobium, boron, yttrium, palladium, and ruthenium, were included in the ILS but the data did not contain the required six laboratories. Precision tables were provided for informational use only.

1.2 The elements and ranges covered in the scope by GD-AES of this test method are listed below.

Element	Tested Mass Fraction Range (%)
Aluminum	0.02 to 7.0
Carbon	0.02 to 0.1
Chromium	0.006 to 0.1
Copper	0.028 to 0.1

Element	Tested Mass Fraction Range (%)
Iron	0.09 to 0.3
Molybdenum	0.016 to 0.1
Nickel	0.006 to 0.1
Silicon	0.018 to 0.1
Tin	0.022 to 0.1
Vanadium	0.054 to 5.0
Zirconium	0.026 to 0.1

1.2.1 The elements boron, manganese, oxygen, nitrogen, niobium, yttrium, palladium, and ruthenium were included in the ILS, but the data did not contain the required six laboratories. Precision tables were provided for informational use only.

1.3 The elements and mass fractions given in the above scope tables are the ranges validated through the interlaboratory study. However, it is known that the techniques used in this standard allow the useable range, for the elements listed, to be extended higher or lower based on individual instrument capability, available reference materials, laboratory capabilities, and the spectral characteristics of the specific element wavelength being used. It is also acceptable to analyze elements not listed in 1.1 or 1.2 and still meet compliance to this standard test method. Laboratories must provide sufficient evidence of method validation when extending the analytical range or when analyzing elements not reported in Section 18 (Precision and Bias), as described in Guide E2857.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Specific safety hazard statements are given in Section 9.*

1.5 *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.*

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 E01.06 on Ti, Zr, W, Mo, Ta, Nb, Hf, Re.

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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
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E305 Practice for Establishing and Controlling Spark Atomic Emission Spectrochemical Analytical Curves
- E406 Practice for Using Controlled Atmospheres in Atomic Emission Spectrometry
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis (Withdrawn 2019)³
- E1507 Guide for Describing and Specifying the Spectrometer of an Optical Emission Direct-Reading Instrument
- E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
- E2857 Guide for Validating Analytical Methods
- E2972 Guide for Production, Testing, and Value Assignment of In-House Reference Materials for Metals, Ores, and Other Related Materials

2.2 ISO Standard:⁴

- ISO/IEC Guide 98-3:2008 Uncertainty of Measurement—Part 3: Guide to the Expression of Uncertainty in Measurement (GUM:1995)—First Edition

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *alloy-type calibration, n*—calibrations determined using reference materials from titanium alloys with generally similar compositions.

3.2.2 *global type calibration, n*—calibrations determined using reference materials from numerous different titanium alloys with considerable compositional variety.

3.2.3 *type standardization, n*—mathematical adjustment of the calibration curve's slope or intercept, or both, using a single reference material at or close to the nominal composition for the particular alloy being analyzed. For best results, the reference material being used should be of the same alloy family as the material being analyzed.

4. Summary of Test Method

4.1 *Spark-AES*—A controlled electrical discharge is produced in an argon atmosphere between the prepared flat surface

of a specimen and the tip of a counter electrode. The energy of the discharge is sufficient to ablate material from the surface of the specimen, break the chemical or physical bonds, and cause the resulting atoms or ions to emit radiant energy. The radiant energies of the selected analytical lines and the internal standard line(s) are converted into electrical signals by either photomultiplier tubes (PMTs) or a suitable solid-state detector. The detected analyte signals are integrated and converted to an intensity value. A ratio of the detected analyte intensity and the internal standard signal may be made. A calibration is made using a suite of reference materials with compositional similarity to the specimens being analyzed. Calibration curves plotting analyte intensity (intensity ratio) versus analyte mass fraction are developed. Specimens are measured for analyte intensity and results in mass fraction are determined using the calibration curves.

4.2 *GD-AES*—A glow discharge lamp creates a low-pressure Ar plasma above the sample surface by applying a high negative voltage between the sample (cathode) and an anode. Argon ions are accelerated into the specimen, which sputters material from the surface. The sputtered material diffuses into the argon plasma where it is dissociated into atoms and excited. The light emitted from these excited species is characteristic of the elements composing the sample and is converted into electrical signals by either photomultiplier tubes (PMTs) or a suitable solid-state detector. The detected analyte signals are integrated and converted to an intensity value. A ratio of the detected analyte intensity and the internal standard signal may be made. A calibration is made using a suite of reference materials with compositional similarity to the specimens being analyzed. Calibration curves plotting analyte intensity (intensity ratio) versus analyte mass fraction are developed. Specimens are measured for analyte intensity and results in mass fraction are determined using the calibration curves.

5. Significance and Use

5.1 This test method for the chemical analysis of titanium alloys is primarily intended to test material for compliance to compositional requirements of specifications such as those under jurisdiction of ASTM Committee B10. It may also be used to test compliance with other specifications that are compatible with the test method.

5.2 This is a performance-based test method that relies more on the demonstrated quality of the test result than on strict adherence to specific procedural steps. It is assumed that all who use this test method will be trained analysts capable of performing common laboratory procedures skillfully and safely, and that the work will be performed in a properly equipped laboratory.

5.3 It is expected that laboratories using this test method will prepare their own work instructions. These work instructions will include detailed operating instructions for the specific laboratory, the specific reference materials employed, and performance acceptance criteria.

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.

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandinnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.