



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

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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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.*

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, <https://www.iso.org>.

6. Recommended Analytical Lines and Potential Interferences

6.1 In Spark-AES or GD-AES atomic emission, when possible, select analytical lines which are free from spectral interferences. However, this is not always possible, and it may be necessary to apply background or inter-element corrections to account mathematically for the effect of the interference on the measured intensities. If interference corrections are necessary, refer to Practice E305 for detailed information on the various techniques used to calculate interference corrections.

6.2 Table 1 lists analytical lines routinely used for analysis of titanium alloys. For consistency of expression, the wavelengths are all listed as stated in the National Institute of Standards and Technology (NIST) Atomic Spectroscopy Database. In the NIST wavelength table, wavelengths < 200 nm are as determined in a vacuum and wavelengths ≥ 200 nm are as determined in air. Potential spectral interferences are also indicated. It is not implied that measurements for this standard test method must be made under the analytical conditions used by NIST. Refer to Section 7 for a discussion of appropriate spectrometer configurations.

7. Apparatus

7.1 Excitation Source:

7.1.1 Spark Source, unipolar, triggered capacitor discharge. In today's instrumentation, the excitation source is computer controlled and is normally programmed to produce: (1) a high-energy pre-spark (of some preset duration), (2) a spark-type discharge (of some preset duration), (3) an arc type discharge (of some preset duration), and (4) a spark-type discharge, during which, time resolved measurements are made for improved detection limits (this may be optional on some instruments).

7.1.2 Glow Discharge Source, capable of producing an argon plasma discharge. With current instrumentation, the excitation source may be direct current (DC) or radio frequency (RF) based.

7.2 Gas Flow System—Designed to deliver pure argon gas to the excitation/sample interface region. Use the minimum argon purity specified by the instrument manufacturer. Refer to Practice E406 for practical guidance on the use of controlled atmospheres.

7.3 Spectrometer—Having acceptable dispersion, resolution, and wavelength coverage for the determination of titanium alloys. As described in Guide E1507.

7.4 Optional Optical Path Purge or Vacuum System—Designed to enhance vacuum wavelength sensitivity by either purging the optical path with a UV-transparent gas or by evacuating the optical path to remove air. The UV-transparent gas must meet the manufacturer's minimum suggested purity requirements.

7.5 Measuring and Control Systems—Designed to convert emitted light intensities to a measurable electrical signal. These systems will consist of either a series of photomultiplier tubes (PMT) or solid-state photosensitive arrays ((Charge Coupled

TABLE 1 Analytical Lines for the Analysis of Titanium Alloys and Potential Interferences

Elements	Wavelength, λ (nm)	Potential Interferences, λ (nm)
Aluminum	236.70	
	256.799	Zr 256.764
	394.401	
	396.152	
Boron	182.64	
	208.957	
	249.678	Fe 249.678
Carbon	165.701	
	165.812	
	193.027	Al 193.041
Chromium	284.325	Zr 284.352
	425.433	
Copper	200.3	
	327.396	
	510.554	
Iron	371.993	
	259.940	Ti 259.992
	259.957	
Manganese	293.31	
	403.076	
	403.307	
	403.449	
Molybdenum	202.02	
	290.91	
	386.411	Zr 386.387
Nickel	341.476	Zr 341.466
	231.604	
	316.34	W 316.342
Niobium	319.50	
	405.89	
	149.26	
Nitrogen	174.272	
	130.22	
Oxygen	340.458	Mo 340.434, Zr 340.483
	363.470	
Palladium	349.894	
	372.803	
Ruthenium	212.415	
	251.611	
	288.158	Cr 288.123
Silicon	140.0454	
	147.5	
	189.989	
Tin	303.41	
	317.505	Fe 317.544
	337.279	
Titanium	367.16	
	374.16	
	239.71	Zr 429.479
Tungsten	429.461	
	214.01	
	326.770	
Vanadium	411.179	W 411.182
	437.924	Zr 437.978
	360.073	Zr 360.119
Yttrium	371.029	Ti 370.996
	339.198	Fe 339.23, Nb 339.234
	343.823	
Zirconium	357.247	Fe 357.200, W 357.240
	360.119	Cr 360.167
	306.77	
Bismuth ^A	165.70	
Carbon ^A	228.62	
Cobalt ^A	383.05	
Europium ^A	227.33	
Hafnium ^A	296.33	
Tantalum ^A	239.71	
Tungsten ^A		

^A Suggested wavelength as data for the analyses of these elements by this test method is very limited.