



# Standard Test Method for Analysis of Titanium and Titanium Alloys by Direct Current Plasma and Inductively Coupled Plasma Atomic Emission Spectrometry (Performance-Based Test Methodology)<sup>1</sup>

This standard is issued under the fixed designation E2371; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This method describes the analysis of titanium and titanium alloys, such as specified by committee B10, by inductively coupled plasma atomic emission spectrometry (ICP-AES) and direct current plasma atomic emission spectrometry (DCP-AES) for the following elements:

Element	Application Range (wt.%)	Quantitative Range (wt.%)
Aluminum	0–8	0.009 to 8.0
Boron	0–0.04	0.0008 to 0.01
Cobalt	0–1	0.006 to 0.1
Chromium	0–5	0.005 to 4.0
Copper	0–0.6	0.004 to 0.5
Iron	0–3	0.004 to 3.0
Manganese	0–0.04	0.003 to 0.01
Molybdenum	0–8	0.004 to 6.0
Nickel	0–1	0.001 to 1.0
Niobium	0–6	0.008 to 0.1
Palladium	0–0.3	0.02 to 0.20
Ruthenium	0–0.5	0.004 to 0.10
Silicon	0–0.5	0.02 to 0.4
Tantalum	0–1	0.01 to 0.10
Tin	0–4	0.02 to 3.0
Tungsten	0–5	0.01 to 0.10
Vanadium	0–15	0.01 to 15.0
Yttrium	0–0.04	0.001 to 0.004
Zirconium	0–5	0.003 to 4.0

1.2 This test method has been interlaboratory tested for the elements and ranges specified in the quantitative range part of the table above. It may be possible to extend this test method to other elements or broader mass fraction ranges as shown in the application range part of the table above provided that test method validation is performed that includes evaluation of method sensitivity, precision, and bias. Additionally, the validation study shall evaluate the acceptability of sample preparation methodology using reference materials or spike recoveries or both. Guide E2857 provides information on validation of analytical methods for alloy analysis.

<sup>1</sup> 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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1.3 Because of the lack of certified reference materials (CRMs) containing bismuth, hafnium, and magnesium, these elements were not included in the scope or the interlaboratory study (ILS). It may be possible to extend the scope of this test method to include these elements provided that method validation includes the evaluation of method sensitivity, precision, and bias during the development of the testing method.

1.4 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.5 *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 safety hazards statements are given in Section 9.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

D1193 Specification for Reagent Water

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E1097 Guide for Determination of Various Elements by Direct Current Plasma Atomic Emission Spectrometry

E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis

E1479 Practice for Describing and Specifying Inductively-Coupled Plasma Atomic Emission Spectrometers

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods

<sup>2</sup> 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.

**E1832 Practice for Describing and Specifying a Direct Current Plasma Atomic Emission Spectrometer**

**E2027 Practice for Conducting Proficiency Tests in the Chemical Analysis of Metals, Ores, and Related Materials**

**E2626 Guide for Spectrometric Analysis of Reactive and Refractory Metals**

**E2857 Guide for Validating Analytical Methods**

2.2 *ISO Standards*:<sup>3</sup>

**ISO/IEC 17025 Requirements for the Competence of Calibration and Test Laboratories**

**ISO Guide 31 Reference Materials—Contents of Certificates and Labels**

**ISO Guide 34 General Requirements for the Competence of Reference Material Producers**

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

### 3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology **E135**.

### 4. Summary of Test Method

4.1 A mineral acid solution of the sample is aspirated into an inductively coupled plasma (ICP) or direct current plasma (DCP) spectrometer. The intensities of emission lines from the spectra of the analytes are measured and compared with calibration curves obtained from solutions containing known amounts of pure elements.

### 5. Significance and Use

5.1 This test method for the chemical analysis of titanium and titanium alloys is primarily intended to test material for compliance with specifications of chemical composition such as those under the 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 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 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 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 used, and performance acceptance criteria. It is also expected that, when applicable, each laboratory will participate in proficiency test programs, such as described in Practice **E2027**, and that the results from the participating laboratory will be satisfactory.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

## 6. Interferences

6.1 In Practice **E1479**, the typical interferences encountered during inductively coupled plasma spectrometric analysis of metal alloys are described. In Guide **E1097**, the typical interferences encountered during direct current plasma emission spectrometric analysis of metal alloys are described. The user is responsible for ensuring the absence of, or compensating for, interferences that may bias test results obtained using their particular spectrometer.

6.2 The use of an internal standard may compensate for the physical interferences resulting from differences between sample and calibration solutions transport efficiencies.

6.3 Shifts in background intensity levels because of recombination effects or molecular band contributions or both may be corrected by the use of an appropriate background correction technique. Direct spectral overlaps are best addressed by selecting alternative wavelengths. Spectral interference studies should be conducted on all new matrices to determine the interference correction factor(s) that shall be applied to concentrations obtained from certain spectral line intensities to minimize biases. Some instrument manufacturers offer software options that mathematically correct for direct spectral overlaps, but the user is cautioned to evaluate carefully this approach to spectral correction.

6.4 Modern instruments have software that allows comparison of a sample spectrum to the spectrum obtained from a blank solution. The user of this test method shall examine this information to ascertain the need for background correction and the correct placement of background points.

6.5 In **Table 1**, wavelengths that may be used for analysis of titanium alloys are suggested. Each line was used by at least one laboratory during the interlaboratory phase of test method development and provided statistically valid results. Additional elements and wavelengths may be added if proficiency is demonstrated. Information for the suggested analytical wavelengths was collected from each laboratory and has been converted to wavelengths as annotated in the NIST Atomic Spectra Database.<sup>4</sup> In this database, wavelengths of less than 200 nm were measured in vacuum and wavelengths greater than or equal to 200 nm were measured in air. Additionally, the MIT Wavelength Tables<sup>5</sup> were used. Tables for individual instruments may list wavelengths somewhat differently, as instrument optical path atmospheric conditions may vary.

6.6 Information on potential spectral interfering elements was provided by the laboratories participating in the interlaboratory study (ILS) and may have originated from sources such as recognized wavelength reference tables, instrument manufacturer's software wavelength tables, and/or an individual laboratory's wavelength research studies.

<sup>4</sup> Ralchenko, Yu., Kramida, A. E., Reader, J., and NIST ASD Team, *NIST Atomic Spectra Database* (version 3.1.5), 2008, online. Available: <http://physics.nist.gov/asd3> [2008, October 28]. National Institute of Standards and Technology, Gaithersburg, MD.

<sup>5</sup> Harrison, G. R., *MIT Wavelength Tables*, John Wiley & Sons, New York, New York, 1969.

**TABLE 1 Analytical Lines and Potential Interferences**

Element	Wavelength (nm)	Potential Interference
Aluminum	176.639	
Aluminum	394.400	
Bismuth (see 1.4)	190.241	
Boron	182.579	Molybdenum, cobalt, chromium
Boron	249.678	Tin, chromium, iron
Boron	208.893	
Cobalt	230.786	
Cobalt	231.160	Antimony, nickel
Cobalt	235.342	
Cobalt	237.863	Iron
Cobalt	238.89	
Copper	224.701	
Copper	327.396	
Chromium	267.716	
Chromium	206.553	Tungsten
Chromium	266.602	Cobalt
Chromium	275.072	Iron, molybdenum
Hafnium (see 1.4)	277.336	
Hafnium (see 1.4)	232.247	
Iron	261.187	
Iron	259.940	
Magnesium (see 1.4)	280.270	
Manganese	257.611	Cerium, cobalt, tungsten
Manganese	260.568	
Molybdenum	201.510	
Molybdenum	202.030	
Nickel	231.604	
Niobium	288.318	
Niobium	295.088	Hafnium
Palladium	340.458	
Palladium	355.308	
Palladium	360.955	
Ruthenium	240.272	
Ruthenium	245.553	
Silicon	251.611	Hafnium, molybdenum
Silicon	288.160	Chromium
Tantalum	240.062	Iron
Tin	175.791	
Tin	242.949	
Titanium Internal Standard	191.391	
Titanium Internal Standard	247.417	
Titanium Internal Standard	326.369	
Titanium Internal Standard	348.966	
Titanium Internal Standard	358.713	
Titanium Internal Standard	372.459	
Titanium Internal Standard	431.506	
Tungsten	207.911	
Vanadium	292.402	Iron, molybdenum
Vanadium	326.770	
Vanadium	354.519	Niobium, tungsten
Vanadium	359.202	
Yttrium	360.073	Molybdenum
Zirconium	343.823	Nickel

6.7 The user shall verify that the selected wavelength performs acceptably in their laboratory, preferably during method validation (See Section 15). The user also may choose to use multiple wavelengths to help verify that line selection is optimized for the particular alloy being determined. It is recommended that when wavelengths and appropriate spectral corrections are determined, the user of this test method should

either specify this information or reference instrument programs that include this information in their laboratory analysis procedures.

## 7. Apparatus

7.1 Direct current plasma atomic emission spectrometers (DCP-AES) used in this test method may conform to the specifications given in Practice E1832. A differently designed instrument may provide equivalent measurements. Suitability for use is determined by comparing the results obtained with the precision and bias statements contained in this method.

7.2 Inductively coupled plasma atomic emission spectrometers (ICP-AES) used in this test method may conform to the specifications given in Practice E1479. A differently designed instrument may provide equivalent measurements. Suitability for use is determined by comparing the results obtained with the precision and bias statements contained in this test method.

7.3 The sample introduction system shall be constructed of materials resistant to all mineral acids including hydrofluoric acid (HF).

7.4 Each instrument shall be set up according to the manufacturer's instructions.

7.5 Machine tools capable of removing surface oxides and other contamination from the as-received sample may be used to produce uncontaminated and chemically representative chips or millings for analysis.

## 8. Reagents and Materials

### 8.1 Reagents:

8.1.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>6</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water is sufficiently purified to meet the requirements of Type II of Specification D1193 or equivalent. The water purification method used shall be capable of removal of all elements in concentrations that might bias the test results.

8.1.3 *Internal Standard*—The use of an internal standard is optional. However, the use of an internal standard may compensate for the physical interferences resulting from differences in sample and calibration solutions transport efficiency. They also help compensate for daily instrumental drift as a result of temperature changes and so forth.

### 8.2 Calibration Solutions:

8.2.1 In this test method, calibration is based on laboratory prepared, alloy matrix-matched calibration solutions. Alloy

<sup>6</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.