



Designation: E2371 – 04

Standard Test Method for Analysis of Titanium and Titanium Alloys by Atomic Emission Plasma Spectrometry¹

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1. Scope

1.1 This method describes the analysis of titanium and titanium alloys by ICP-AES (Inductively Coupled Plasma) and DCP-AES (Direct Current Plasma) for the following elements:

Element	Application Range (wt.%)	Quantitative Range (wt.%)
Aluminum	0–8	0.001 to 8.0
Boron	0–0.04	0.0008 to 0.01
Chromium	0–5	0.005 to 4.0
Copper	0–0.6	0.002 to 0.5
Iron	0–3	0.004 to 3.0
Manganese	0–0.04	0.001 to 0.01
Molybdenum	0–8	0.004 to 6.0
Nickel	0–1	0.001 to 1.0
Silicon	0–0.5	0.02 to 0.4
Tin	0–4	0.02 to 3.0
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 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:²

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

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

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 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.

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

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

E1914 Practice for Use of Terms Relating to the Development and Evaluation of Methods for Chemical Analysis

3. Terminology

3.1 For definitions of terms used in this method, refer to Terminology **E135** and Terminology section in **E1914**.

4. Summary of Test Method

4.1 A mineral acid solution of the sample is aspirated into an inductively coupled plasma or a direct current plasma 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 method for the chemical analysis of titanium and titanium alloys is primarily intended to test material for compliance with specifications of chemical composition.

5.2 It is assumed that all who use this 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 The method is designed to give the maximum flexibility analyzing elements in the titanium matrix. Thus options are given in calibration and analysis to accommodate the variety of ICP and DCP spectrometers and their auxiliary systems.

6. Interferences

6.1 Potential interferences for analytes (see **1.2**) are listed in **Table 1** for this method's analytical wavelengths. The analytical wavelengths of **Table 1** were selected for their freedom from interference by the elements included in this method and the titanium base (see **Note 1**).

TABLE 1 Analytical Lines and Potential Interferences

Element	Wavelength (nm)	Potential Interferences	Notes
Al	394.401		
B	249.678		
Cr	267.716		
Cu	327.396		
Fe	259.940		
Mn	257.610		
Mo	202.030		
Ni	231.604		
Si	288.160	Cr	
Sn	175.790		2
	242.949	Mo, Fe	3
V	292.402	Mo, Fe	
Y	360.073	Mo	
Zr	343.823		

Note—Sn 175.790 – Low UV Capable

Sn 242.949 – DCP; non-Low UV capable alternative

NOTE 1—Elements outside the scope of this method may be present in production materials or experimental titanium alloys. These potential interferences must be tested for their effect as an interference on analyte wavelengths and corrections made as necessary as a part of method development according to manufacturer's instructions.

7. Apparatus

7.1 Direct Current Plasma Atomic Emission Spectrometers used in this 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 used in this 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 method.

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

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

8. Reagents and Materials

8.1 Purity of reagents utilized in this procedure shall conform to the requirements prescribed in Practice E50.

8.2 The purity of water used shall conform to the requirements in Specification D1193 for reagent water, Type II.

8.3 The argon supply for the ICP or DCP plasma shall be of 99.998 % minimum purity; for purging the ICP Optical path, 99.999 % minimum purity.

8.4 The nitrogen supply for purging the ICP Optical path shall be 99.995 % minimum purity.

8.5 Reference solutions are available for purchase as an alternative to the preparation of solutions from pure metals or compounds.

8.5.1 Single element reference solutions in the form of Certified Reference Materials are available from the National Institute of Standards and Technology (NIST) and a number of

commercial suppliers. The NIST Standard Reference Materials (SRM) 3100³ series are available as 10 mg/g (5 mg/g for some elements) solutions. See Notes 2 and 3.

8.5.2 Reference solutions from other sources may be used if they have a documented link of traceability to primary reference materials or CRMs from a national metrology institute and the concentrations of impurities are known or determined (see Note 4).

NOTE 2—To use solution standards of concentration less than the original concentration, prepare the solution by dilution of the concentrated reference solution. Maintain the acid concentration as recommended in the NIST or other certificate. NIST certificates provide instructions for dilution by mass or by volume. Dilution by mass may be preferable to pipetting, especially for solutions containing HF.

NOTE 3—The user should establish internal laboratory procedures that specify a maximum shelf life for a working standard solution. It has been observed that polytetrafluoroethylene (PTFE) bottles preserve the integrity of the solutions stored in them for a long period of time.

NOTE 4—If single element solutions are not provided with values for trace impurities, it is necessary to either determine the concentrations of elements that are in the scope of this method or, if they cannot be detected, ensure that the method detection limit is sufficiently low that the impurities cannot be detected in solutions prepared for calibration of this method.

8.5.3 *Calibrants Prepared from Pure Metals or their Salts* (see Notes 5 and 6):

NOTE 5—Elemental solutions of 10.0 mg/mL or 1.00 mg/mL can be stored in HDPE or polytetrafluoroethylene (PTFE) for one (1) year. The 0.100 mg/mL solutions must be made fresh before each use.

NOTE 6—Elements and compounds for the solutions below are usually supplied on a "metals basis" assay. The concentration of the element sought, as percent in the present usage, is the total metallic content of the compound less the assayed metallic impurities (including B, Si, Y).

8.5.3.1 *Aluminum Standard Solution A* (1 mL = 10.0 mg Al)—Dissolve 10.000 g aluminum wire (purity 99.99 %, min) in 200 mL of HCl (1+1) with gentle heating. Cool, transfer to a 1 L volumetric flask, dilute to the mark and mix. (A small crystal of mercuric nitrate may be added to catalyze the dissolution reaction.)

8.5.3.2 *Boron Standard Solution A* (1 mL = 1.00 mg B)—Dissolve 5.720 g of boric acid, H₃BO₃ (purity 99.99 %, min) in 500 mL of water. Transfer to a 1 L volumetric flask, dilute to the mark and mix. Store in a plastic bottle.

8.5.3.3 *Chromium Standard Solution A* (1 mL = 10.0 mg Cr)—Dissolve 10.00 g of chromium (purity 99.99 %, min.) chips in 200 mL of HCl (1+1) with gentle heating. Cool, transfer to a 1 L volumetric flask, dilute to the mark and mix.

8.5.3.4 *Copper Standard Solution A* (1 mL = 10.0 mg Cu)—Dissolve 10.00 g of copper wire (purity 99.99 %, min.) in 100 mL of HNO₃ (1+1) with gentle heating. Continue heating until brown fumes cease to evolve. Cool, transfer to a 1 L volumetric flask, and add 50 mL of HNO₃, dilute to the mark and mix.

8.5.3.5 *Copper Standard Solution B* (1 mL = 1.00 mg Cu)—Pipet 10 mL of Copper Standard Solution A into a 100 mL volumetric flask, add 10 mL of HNO₃, dilute to the mark and mix.

³ Available from National Institute of Standards and Technology, 100 Bureau Drive, Gaithersburg, MD 20899, USA.

8.5.3.6 Iron Standard Solution A (1 mL = 10.0 mg Fe)—Dissolve 10.00 g of iron rod (purity 99.99 %, min, that has been cleaned to remove oxidation) in 100 mL of HNO₃ by heating to boiling. Continue gentle boiling until brown fumes cease to evolve. Cool, transfer to a 1 L volumetric flask, dilute to the mark and mix.

8.5.3.7 Manganese Standard Solution A (1 mL = 1.00 mg Mn)—Dissolve 1.000 g of manganese (purity 99.98 %, min) in 100 mL of HNO₃ (1+1) with gentle heating. Boil gently to expel brown fumes and cool. Transfer to a 1 L volumetric flask, add 50 mL of HNO₃, dilute to the mark and mix.

8.5.3.8 Molybdenum Standard Solution A (1 mL = 10.0 mg Mo)—Transfer 5.00 g of molybdenum rod (purity 99.98 %, min) to a 600 mL beaker. Add 200 mL of an acid mixture of HCl, HNO₃ and water (3+2+1) and heat gently to dissolve. Cool, transfer to a 500 mL volumetric flask, dilute to the mark and mix.

8.5.3.9 Nickel Standard Solution A (1 mL = 10.0 mg Ni)—Dissolve 10.00 g of nickel wire (purity 99.99 %, min) in 100 mL of HNO₃ with gentle heating. Continue heating until brown fumes are no longer evolved. Cool, transfer to a 1 L volumetric flask, dilute to the mark and mix.

8.5.3.10 Nickel Standard Solution B (1 mL = 1.00 mg Ni)—Pipet 10 mL of Nickel Standard Solution A into a 100 mL volumetric flask, add 10 mL of HNO₃, dilute to the mark and mix.

8.5.3.11 Silicon Standard Solution A (1 mL = 1.00 mg Si)—Fuse 0.2139 g of silicon dioxide (purity 99.99 %, min) with 2.00 g of sodium carbonate (purity 99.995 %, min) in a platinum crucible. Dissolve the melt in water, transfer to a 100 mL flask, dilute to the mark and mix. Store in a plastic bottle.

8.5.3.12 Tin Standard Solution A (1 mL = 10.0 mg Sn)—Dissolve 10.00 g of tin wire (purity 99.99 %, min) in 100 mL of HCl with gentle heating. Cool, transfer to a 1 L volumetric flask, and add 400 mL of HCl, dilute to the mark and mix.

8.5.3.13 Vanadium Standard Solution A (1 mL = 10.0 mg V)—Dissolve 17.852 g of vanadium (V) oxide (V₂O₅) (purity 99.99 %, min) in a 1 L volumetric flask by adding 300 mL of HCl and warm (≤ 75°C) to complete dissolution. Cool, dilute to the mark and mix.

8.5.3.14 Yttrium Standard Solution A (1 mL = 1.00 mg Y)—Dissolve 1.270 g of yttrium oxide (Y₂O₃) (purity 99.99 %, min) in 30 mL of HCl (1+1). Transfer to a 1 L volumetric flask, dilute to the mark and mix.

8.5.3.15 Zirconium Standard Solution A (1 mL = 10.0 mg Zr)—Transfer 35.326 g of ZrOCl₂ · 8H₂O (purity 99.99 %, min) to a 1 L volumetric flask, add approximately 500 mL of water, and swirl until dissolved. Add 50 mL of HCl, cool, dilute to the mark and mix.

8.6 Titanium base metal for the production of all calibration standards shall be of 99.99 % minimum purity on a metals basis. See [Notes 6 and 7](#).

NOTE 7—Impurities of this titanium base material shall be determined by Glow Discharge Mass Spectrometry (GDMS) or suitable technique that has detection limits for each respective analyte that is less than that of the ICP or DCP in the titanium matrix.

8.7 An internal standard may be used to control drift. If an internal standard is used, it shall not interfere with the analytes of this method.

8.8 A surfactant may be used to control droplet formation in the spray chamber and sample tubes of the DCP spectrometer. A maximum 0.1 % Vol/Vol of a surfactant such as Polyoxyethylene(10)isooctylphenyl ether is recommended.

8.9 Calibration Solutions:

8.9.1 Standard Solution A:

8.9.1.1 Add 5.00 g (ICP system) or 10.00 g (DCP system) of titanium (purity 99.99 %, min) to a 1 L plastic volumetric flask (see [Note 8](#)).

NOTE 8—The ICP procedure and DCP procedure require differing sample sizes. This is maintained consistently throughout the method in terms of sample weights and analyte additions for samples, calibration solutions and quality control solutions.

8.9.1.2 Add 150 mL of HCl, then 20 mL of HF (see warning note below in [8.9.1.4](#)).

8.9.1.3 After all titanium has dissolved, add 10 mL of HNO₃ (ICP) or 15 mL of HNO₃ (DCP) to oxidize.

8.9.1.4 Cool, add internal standard solution and/or surfactant if desired, dilute to the mark and mix. (**Warning**—HF causes burns that require immediate medical attention even though they are not immediately painful; refer to the paragraph about HF in the Safety Precautions of Practice [E50](#).)

8.9.1.5 Store in a polytetrafluoroethylene (PTFE) bottle. Label the bottle and mark with the date of preparation. This solution's shelf life is six (6) months.

8.9.1.6 Adjust the “0” values of Standard A to reflect the actual concentration of the analytes in the titanium base material before calibration.

8.9.2 Standard Solutions B-G:

8.9.2.1 Weigh quantities of titanium (purity 99.99 % min) into 1 L plastic volumetric flasks in accordance with [Appendix X1](#) for ICP and [Appendix X2](#) for DCP.

8.9.2.2 To each, add the specified volumes of HCl and HF (see warning in [8.9.1.4](#)) in accordance with [Appendix X1](#) for ICP and [Appendix X2](#) for DCP.

8.9.2.3 After the titanium dissolves completely, add the specified volumes of HNO₃ in accordance with [Appendix X1](#) for ICP and [Appendix X2](#) for DCP.

8.9.2.4 Cool, add volumes of standard solutions in accordance with [Appendix X1](#) for ICP and [Appendix X2](#) for DCP.

8.9.2.5 Add internal standard solution and/or surfactant if desired. Dilute to volume and mix. Store in a polytetrafluoroethylene (PTFE) bottle. Shelf life is three (3) months.

8.9.2.6 Adjust the concentration values of Standard Solutions B-G to reflect the actual concentration of the analytes in the titanium base material before calibration.

8.9.3 Standard Solution H (Optional):

8.9.3.1 Some instrument systems require more data points for optimum calibration, not only in the range of the major alloy elements but also the trace elements as their concentration approaches zero. An additional calibration material can be used to satisfy this requirement (see [Note 9](#)).

8.9.3.2 This calibration reference material can be a “matrix-matched” solution which contains the major alloying elements (additions of the solutions of section [8.5.3](#)) and the traces of the analytes (made from additions of serial dilutions of the solutions of paragraph [8.5.3](#)) added to the appropriate weight of titanium base. The solution is then