
**6Al-4V titanium alloys —
Determination of aluminium and
vanadium contents — Inductively
coupled plasma atomic emission
spectrometric method**

*6Al-4V alliages de titane — Détermination de la teneur en aluminium
et en vanadium — Méthode par spectrométrie d'émission atomique
avec plasma à couplage inductif*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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6Al-4V titanium alloys — Determination of aluminium and vanadium contents — Inductively coupled plasma atomic emission spectrometric method

1 Scope

This document specifies an inductively coupled plasma atomic emission spectrometric method for the determination of the contents (mass fraction) of aluminium and vanadium in 6Al-4V titanium alloys.

This method is applicable to all kinds of 6Al-4V titanium alloys specified in ISO 23515 (designation of titanium alloys) for aluminium in the range from 4,70 % to 7,00 % and vanadium in the range from 3,00 % to 5,00 %.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Principle

A test portion is dissolved with nitric and hydrofluoric acids. After suitable dilution and, if necessary, addition of an internal standard element, the solution is nebulized into an inductively coupled plasma atomic emission spectrometer and the intensity of the emitted light from each element is measured (including, where relevant, the intensity of the internal standard element).

5 Reagents

5.1 General

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696.

5.2 Hydrochloric acid solution, 1+1

Add slowly 500 ml of hydrochloric acid (ρ_{20} 1,16 g/ml to 1,19 g/ml) to 500 ml of water and mix.

5.3 Nitric acid solution, 1+1

Add slowly 500 ml of nitric acid (ρ_{20} 1,42 g/ml) to 500 ml of water and mix.

5.4 Hydrofluoric acid solution, 1+1

Add, carefully and slowly, 100 ml of hydrofluoric acid (ρ_{20} 1,14 g/ml) to 100 ml of water and mix.

5.5 Titanium

High purity titanium [min 99,9 % (mass fraction)], containing less than or equal to 0,005 % (mass fraction) of aluminium and vanadium.

5.6 Indium solution, 1 g/l

Weigh 1,000 g of high purity indium [min 99,9 % (mass fraction)]. Transfer it into a 300 ml beaker and add 100 ml of hydrochloric acid (5.2) and 20 ml of nitric acid (5.3). Heat gently to complete dissolution. Cool, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

5.7 Cobalt solution, 1 g/l

Weigh 1,000 g of high purity cobalt [min 99,9 % (mass fraction)]. Transfer it into a 300 ml beaker and add 100 ml of nitric acid (5.3). Heat gently to complete dissolution. Cool, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

5.8 Strontium solution, 1 g/l

Weigh 3,043 g of strontium chloride [$\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$]. Transfer it into a 300 ml beaker. Dissolve with water, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

5.9 Aluminium standard solution, 1 g/l

Weigh 1,000 g of high purity aluminium [min 99,9 % (mass fraction)]. Transfer it into a 300 ml beaker and add 100 ml of hydrochloric acid (5.2) and 20 ml of nitric acid (5.3). Heat gently to complete dissolution. Cool, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of aluminium.

5.10 Vanadium standard solution, 1 g/l

Weigh 1,000 g of high purity vanadium [min 99,9 % (mass fraction)]. Transfer it into a 300 ml beaker and add 100 ml of hydrochloric acid (5.2) and 20 ml of nitric acid (5.3). Heat gently to complete dissolution. Cool, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of vanadium.

6 Apparatus

6.1 General

The usual laboratory apparatus and, in particular, the following shall be used.

6.2 Volumetric glassware

All volumetric glassware shall be class A, in accordance with ISO 648 or ISO 1042, as appropriate.

6.3 Plastic beaker, volumetric plastic flask and plastic watch glass

Use plastic beakers, volumetric plastic flasks and plastic watch glasses.

6.4 Atomic emission spectrometer, equipped with an inductively coupled plasma (ICP-AES)

The instrument used shall be suitable for simultaneously measuring the intensity of the emission lines of the analytes and that of the internal standard.

The spectrometer can be either a simultaneous or a sequential one. If a sequential spectrometer can be equipped with an extra arrangement for simultaneous measurement of the internal standard line, it can be used with the internal standard method. If the sequential spectrometer is not equipped with this arrangement, an internal standard cannot be used and an alternative measurement technique without internal standard shall be used.

The instrument shall be equipped with a hydrofluoric-acid-resistant nebulizer.

7 Sampling

Sampling and sample preparation shall be carried out by normal agreed procedures. The test sample normally is in the form of millings or drillings and no further preparation of the sample is necessary.

Millings or drillings shall be uniform in size and not exceed a length of 5 mm. The test samples shall be dry, free from oil, scale and foreign inclusions.

8 Procedure

8.1 Test portion

Weigh to the nearest 0,1 mg, 0,10 g of the test sample.

NOTE The weight of the test portion can vary as long as the user's method demonstrates adequate sensitivity and accuracy.

8.2 Number of determinations

The determinations shall be carried out in duplicate under repeatability conditions.

NOTE For routine purposes and after previous agreement, a single determination can be carried out.

8.3 Preparation of the calibration solutions

Transfer 0,092 g of titanium (5.5) into a series of five 200 ml plastic beakers. Add 10 ml of nitric acid (5.3) and 2 ml of hydrofluoric acid (5.4), cover with a plastic watch glass and heat gently to complete dissolution. Cool to room temperature.

Transfer each solution into a 100 ml volumetric plastic flask. Add to each volumetric flask the volumes of the standard solutions given in Table 1.

Add 5,0 ml of one of the internal standard element solution [indium (5.6), cobalt (5.7) or strontium (5.8)], dilute to the mark with water and mix.