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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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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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This document was prepared by Technical Committee ISO/TC 79, *Light metals and their alloys*, Subcommittee SC 11, *Titanium*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 [$SrCl_2 \cdot 6H_2O$]. 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.

Table 1 — Typical volumes of the standard solutions (ml)

Calibration solution label	Aluminium standard solution (5.9)	Vanadium standard solution (5.10)
T 1	8	0
T 2	6	2
T 3	4	4
T 4	2	6
T 5	0	8

NOTE 1 The use of an internal standard is optional. Indium, cobalt or strontium can be used for this purpose.

NOTE 2 The possible interferences from impurities on determination of aluminium and vanadium contents need to be taken into account when preparing standard solutions.

8.4 Preparation of the test solutions

Transfer the test portion into a 200 ml plastic beaker.

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.

Remove the watch glass and transfer the solution into a 100 ml volumetric plastic flask.

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

NOTE The use of an internal standard is optional. Indium, cobalt or strontium can be used for this purpose.

8.5 Determination

8.5.1 General

Start the ICP-AES and let it stabilize in accordance with the manufacturer's instructions before any measurement.

Optimize the instrument in accordance with the manufacturer's instructions, including the background correction(s) where relevant.

Prepare the software for the measurements of the intensities, and for the calculation of the mean value corresponding to the appropriate analytical line.

Each time the internal standard element is used, prepare the software to calculate the ratio between the intensity of the analyte and the intensity of the internal standard.

8.5.2 Spectrometric measurements of the calibration solutions

Nebulise each calibration solution into the argon plasma of an ICP-atomic emission spectrometer. At the wavelengths shown in Table 2, measure the absolute intensities or the ratioed intensities of the analytical lines.

Table 2 — Element and example of measurement wavelengths

Element	Wavelength nm
Al	394,40, 396,15
V	292,40, 311,07, 309,31, 310,23
In	325,61
Sr	407,77
Co	228,62, 238,89, 230,79

Calculate the intensity ratios of the emission intensities of aluminium, vanadium and the internal standard element using Formula (1).

$$R = \frac{I_X}{I_{IS}} \tag{1}$$

where

R is the intensity ratio of aluminium and the internal standard element or vanadium and the internal standard element;

I_X is the emission intensity of aluminium or vanadium;

I_{IS} is the emission intensity of the internal standard element.

Calculate the linear regression through the points representing the intensities or the ratioed intensities of each element on the y-axis and the corresponding concentration, expressed in mg/100 ml, in the calibration solutions on the x-axis.

Calculate the correlation coefficient of each curve: each coefficient shall be > 0,999.

Once the linearity of the calibration is controlled, recalibrations can be carried out only with a high and low calibration solutions together with an intermediate calibration solution for verification purposes.

8.5.3 Spectrometric measurements of the test solutions

Nebulise each test solution into the argon plasma of an ICP-atomic emission spectrometer.

Measure the absolute intensity or ratioed intensity of the test sample solution.

Determine the aluminium and vanadium concentration (mg/100 ml) of the test solutions from the calibration curve.

It is recommended that a laboratory using this method for the first time should analyse Ti-6Al-V CRMs or RMs to check if this method can be used correctly.

9 Expression of result

The mass fraction of aluminium and vanadium in the test portion, w_x, expressed as a percentage, is given by Formula (2).

$$w_x = \frac{\rho}{m} \times 100 \times 10^{-3} \tag{2}$$

where

ρ is the concentration of aluminium and vanadium in the test solution, expressed in mg/100 ml;

m is the mass of test portion, expressed in g.

Calculate the contents (mass fraction) of aluminium and vanadium in the test portion to the second decimal place.

10 Precision

A planned trial of this method was carried out by nine laboratories, using five levels of aluminium and vanadium contents (see Annex A), each laboratory making three determinations of the aluminium and vanadium contents at each level (see NOTES 1 and 2).

NOTE 1 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725-1, i.e. one operator, same apparatus, identical operating conditions, same calibration and a minimum period of time.

NOTE 2 The third determination was carried out at a different time (on a different day) by the same operator as in NOTE 1, using the same apparatus with a new calibration.

The results obtained were treated statistically according to ISO 5725-2 and ISO 5725-3.

Because of the lower coefficients of correlation of the relations between m and repeatability limit (r), within-laboratory reproducibility limit (R_w) and reproducibility limit (R), only the experimental precision data are presented (see Table 3 and Table 4).

Table 3 — Experimental precision data for aluminium

Aluminium content % mass fraction		**Repeatability limit** r % mass fraction	**Reproducibility limits**	
Certified	Found		R_w % mass fraction	R % mass fraction
4,67	4,629	0,038	0,050	0,107
5,41	5,399	0,110	0,110	0,115
6,20	6,209	0,060	0,100	0,113
6,245	6,229	0,062	0,089	0,153
7,03	7,030	0,046	0,046	0,103

Table 4 — Experimental precision data for vanadium

Vanadium content % mass fraction		**Repeatability limit** r % mass fraction	**Reproducibility limits**	
Certified	Found		R_w % mass fraction	R % mass fraction
5,01	5,059	0,074	0,074	0,161
3,54	3,519	0,061	0,061	0,117
4,02	4,031	0,038	0,046	0,165
4,154	4,160	0,053	0,078	0,120
2,75	2,816	0,044	0,051	0,077

11 Test report

The test report shall include the following information:

a) all information necessary for the identification of the sample, the laboratory and the date of analysis or the test report;

b) the method used with a reference to this document, i.e. ISO 21339:2023;

c) the results and unit in which they are expressed;

d) any unusual features noted during the determinations;

e) any operation not specified in this document, or any optional operation that can possibly have influenced the results.

Annex A
(informative)

Additional information on the international inter-laboratory test

An international inter-laboratory test was carried out in 2020 on 5 Ti-6Al-4V samples in four countries involving nine laboratories.

The composition of the test samples used is listed in Table A.1.

Table A.1 — Composition of test samples used on the international inter-laboratory test

Sample	Chemical composition % mass fraction		
	Al	V	Fe
GBW 02504	4,67	5,01	0,314
France 1	5,41	3,54	0,21
GSB 2404(TC4) 1	6,20	4,02	0,172
France NBS173C	6,245	4,154	0,213
GBW 02508	7,03	2,75	0,073

The graphical representation of the precision data is given in Annex B.