INTERNATIONAL STANDARD

ISO 22962

First edition
2008-01-15

Titanium and titanium alloys — Determination of iron — Inductively coupled plasma atomic emission spectrometry

Titane et alliages de titane — Dosage du fer — Spectrométrie d'émission atomique par plasma à couplage inductif

Reference number
ISO 22962:2008(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

STANDARDSISO.COM : Click to view the full PDF of ISO 22962:2008

COPYRIGHT PROTECTED DOCUMENT

© ISO 2008

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22962 was prepared by Technical Committee ISO/TC 79, *Light metals and their alloys*, Subcommittee SC 11, *Titanium*.

Titanium and titanium alloys — Determination of iron — Inductively coupled plasma atomic emission spectrometry

1 Scope

This International Standard specifies an inductively coupled plasma atomic emission spectrometric method for the determination of the mass fraction of iron in titanium and titanium alloys.

The method is applicable to titanium and titanium alloys with a mass fraction of iron in the range from 0,01 % to 3,0 %.

2 Normative references

The following referenced documents are indispensable for the application 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 384:1978, *Laboratory glassware — Principles of design and construction of volumetric glassware*

ISO 648:—[1], *Laboratory glassware — Single volume pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696:1987, *Water for analytical laboratory use — Specifications and test methods*

ISO 4787:1984, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity*

3 Principle

Dissolve the test portion in nitric acid and hydrofluoric acid, or sulfuric acid and hydrofluoric acid. Determine the iron concentration in the test portion using an inductively coupled plasma (ICP)-atomic emission spectrometer.

4 Reagents

4.1 General

During the analysis, use only reagents of recognized analytical grade and water that complies with grade 2 of ISO 3696.

---

[1] To be published. (Revision of ISO 648:1977)

4.2 Hydrochloric acid (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 (4.1).

4.3 Nitric acid (ρ_{20} 1,42 g/ml)

4.4 Nitric acid (1+1)

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

4.5 Hydrofluoric acid (1+1)

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

4.6 Sulfuric acid (1+1)

Add slowly 500 ml of sulfuric acid (ρ_{20} 1,84 g/ml) to 500 ml of water, while stirring and cooling.

4.7 Sulfuric acid (1+3)

Add slowly 100 ml of sulfuric acid (ρ_{20} 1,84 g/ml) to 300 ml of water, while stirring and cooling.

4.8 Titanium

Minimum purity 99 % (mass fraction), the mass fraction of iron is as low as possible and already known. If the mass fraction of iron is unknown, it should be determined by the mass fraction of iron in ISO 22960.

4.9 Cobalt solution (1 mg Co/ml)

Weigh 1,00 g of cobalt metal [minimum purity 99,5 % (mass fraction)] into a 300 ml beaker, heat and decompose with 40 ml of nitric acid (4.4). After cooling, transfer to a 1 000 ml volumetric flask, make up to the mark with water and mix.

4.10 Yttrium solution (1 mg Y/ml)

Weigh 1,27 g of yttrium oxide(III) [minimum purity 99,5 % (mass fraction)] into a 300 ml beaker, heat and decompose with 20 ml of hydrochloric acid (4.2). After cooling, transfer to a 1 000 ml volumetric flask, make up to the mark with water and mix.

4.11 Lanthanum solution (1 mg La/ml)

Weigh 1,17 g of lanthanum oxide(III) [minimum purity 99,5 % (mass fraction)] into a 300 ml beaker, heat and decompose with 20 ml of hydrochloric acid (4.2). After cooling, transfer to a 1 000 ml volumetric flask, make up to the mark with water and mix.

4.12 Iron standard solution (0,500 mg Fe/ml)

Weigh, to the nearest 0,1 mg, 0,500 g of iron metal [minimum purity 99,9 % (mass fraction)], into a 300 ml beaker, heat and decompose with 30 ml of hydrochloric acid (4.2). Add 5 ml of nitric acid (4.4) to oxidize iron and heat until nitrogen oxides evaporate. After cooling, transfer to a 1 000 ml volumetric flask, make up to the mark with water and mix.

5 Apparatus

5.1 General

Use normal laboratory apparatus.

5.2 Volumetric glassware, of class A complying with ISO 384, ISO 648 and ISO 1042. Use in accordance with ISO 4787.

5.3 Polyethylene beaker and watch glass.

5.4 Polytetrafluoroethylene (PTFE) beaker and **PTFE watch glass**.

5.5 Analytical balance, sensitive to 0,1 mg.

5.6 ICP-atomic emission spectrometer capable of simultaneously measuring the iron emission line 238,20 nm, or 259,94 nm and the emission line of the internal standard cobalt (228,62 nm), yttrium (371,03 nm), lanthanum (398,85 nm), titanium (255,60 nm) or titanium (333,21 nm).

A hydrofluoric-acid-resistant nebulizer is recommended for the decomposition method with nitric acid and hydrofluoric acid (7.3.2).

6 Sample

6.1 Sampling

The sampling procedure for titanium and titanium alloys shall be agreed upon until a corresponding standard method has been published.

6.2 Test portion

Extract a test portion of 0,50 g from the test sample and weigh to the nearest 0,1 mg.

7 Procedure

7.1 Number of determinations

Carry out the determination at least in duplicate, as far as possible under repeatability conditions, on each sample.

7.2 Blank test

Carry out a blank test in parallel with the analysis, using the same quantities of all reagents but omitting the test portion.

7.3 Preparation of calibration solution

7.3.1 General

Prepare the calibration solutions in accordance with 7.3.2, 7.3.3 or 7.3.4.

7.3.2 In the case of decomposition with nitric acid and hydrofluoric acid (This method is used for the procedure described in 7.4.2)

Weigh four samples of titanium (4.8), each of 0,50 g, and transfer to polyethylene beakers (200 ml).

Add 50 ml of nitric acid (4.4) and 10 ml of hydrofluoric acid (4.5), cover with a polyethylene watch glass and heat gently on a water bath to decompose the titanium sample. Continue heating until nitrogen oxides evaporate. Cool to room temperature.

Remove a watch glass, add, from a burette or pipette, 0 ml, 10 ml, 20 ml and 30 ml of iron standard solution (4.12). Transfer the solution into a 100 ml volumetric flask.

Add 5,0 ml of internal standard solution [cobalt (4.9)], make up to the mark with water and mix. Immediately, transfer the solution into a dry polyethylene or PTFE bottle with a stopper and reserve.

7.3.3 In the case of decomposition with sulfuric acid and hydrofluoric acid (This method is used for the procedure described in 7.4.3)

Weigh four samples of titanium (4.8), each of 0,50 g, and transfer to PTFE beakers (200 ml).

Add 20 ml of sulfuric acid (4.6) and 4 ml of hydrofluoric acid (4.5), cover with a PTFE watch glass and heat gently to decompose the titanium. If the decomposition is incomplete, add a small quantity of hydrofluoric acid (4.5) and heat again. Add 4 ml of nitric acid (4.4), and heat for a few minutes.

Remove the watch glass, continue heating until white fumes appear for about 5 min. Then cool to room temperature, and rinse the inside of the beaker with a small quantity of water. Heat again until strong white fumes appear for 2 to 3 min.

The volume of sulfuric acid remaining in the beaker will influence the intensity of emission. Therefore, it is recommended that care be taken to keep a constant volume of the remaining sulfuric acid. However, that influence will be cancelled by using an internal standard method.

Cool to room temperature. Add 20 ml of hydrochloric acid (4.2) and a small quantity of water to dissolve the salt.

Add, from a burette or pipette, 0 ml, 10 ml, 20 ml, and 30 ml of iron standard solution (4.12). Transfer the solution into a 100 ml volumetric flask.

Add 5,0 ml of internal standard solution [cobalt (4.9), yttrium (4.10) or lanthanum (4.11)], make up to the mark with water and mix.

7.3.4 In the case of decomposition with sulfuric acid (This method is used for the procedure in 7.4.4)

Weigh four samples of titanium (4.8), each of 0,50 g, and transfer to conical beakers (100 ml).

Add 40 ml of sulfuric acid (4.7), cover with a watch glass and heat gently to decompose all of the titanium (maintaining the volume with distilled water). After dissolution of the titanium, add nitric acid (4.3) drop by drop for titanium oxidation, and heat the solution until white fumes appear. Cool to room temperature.

Remove a watch glass, add, from a burette or pipette, 0 ml, 10 ml, 20 ml and 30 ml of iron standard solution (4.12). Transfer the solution into a 100 ml volumetric flask with water.

Make up to the mark with water and mix.

7.4 Preparation of a test solution for presentation to the ICP-atomic emission spectrometer

7.4.1 General

Prepare the test solutions for presentation to the ICP-atomic emission spectrometer in accordance with 7.4.2, 7.4.3 or 7.4.4.

7.4.2 Decomposition with nitric acid and hydrofluoric acid

NOTE This method is applicable for the determination of iron in titanium (including commercially available pure titanium) and titanium alloys.

Transfer the test portion to a polyethylene beaker (200 ml).

Add 50 ml of nitric acid (4.4) and 10 ml of hydrofluoric acid (4.5), cover with a polyethylene watch glass and heat gently on a water bath to decompose the test portion. Continue heating until nitrogen oxides evaporate. Cool to room temperature.

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

Add 5,0 ml of internal standard solution [cobalt (4.9)], make up to the mark with water and mix. Immediately, transfer the solution into a dry polyethylene or PTFE bottle with a stopper and reserve.

7.4.3 Decomposition with sulfuric acid and hydrofluoric acid

NOTE This method is applicable for the determination of iron in titanium (including commercially available pure titanium) and titanium alloys.

Transfer the test portion to a PTFE beaker (200 ml).

Add 20 ml of sulfuric acid (4.6) and 4 ml of hydrofluoric acid (4.5), cover with a PTFE watch glass and heat gently to decompose the test portion. If decomposition is incomplete, add a small quantity of hydrofluoric acid (4.5) and heat again. Add 4 ml of nitric acid (4.4) and heat for a few minutes.

Remove the watch glass, continue heating until white fumes appear for 5 min. After cooling to room temperature, rinse the inside of the beaker with a small quantity of water. Heat again until strong white fumes appear for 2 to 3 min.

The volume of the sulfuric acid remaining in the beaker will influence the intensity of emission . Therefore, it is recommended that care be taken to keep a constant volume of the remaining sulfuric acid. However, that influence will be cancelled by using an internal standard method.

Cool to room temperature. Add 20 ml of hydrochloric acid (4.2) and a small quantity of water to dissolve the salt.

After cooling to room temperature, transfer the solution into a 100 ml volumetric flask.

Add 5,0 ml of internal standard solution [cobalt (4.9), yttrium (4.10) or lanthanum (4.11)], make up to the mark with water and mix.

7.4.4 Decomposition with sulfuric acid (using titanium as the internal standard)

NOTE This method uses titanium as the internal standard, and is applicable to the determination of iron in commercially available pure titanium only.

Transfer the test portion to conical beakers (100 ml).

Add 40 ml of sulfuric acid (4.7), cover with a watch glass and heat gently to decompose the titanium (maintaining the volume with distilled water). After dissolution of the titanium, add nitric acid (4.3) drop by drop for titanium oxidation. Heat the solution until white fumes appear.

After cooling to room temperature, transfer the solution into a 100 ml volumetric flask with water.

Make up to the mark with water and mix.

7.5 Determination of iron concentration

7.5.1 General

The instrument shall be set up in accordance with the manufacturer’s instruction. Chose appropriate background correction positions and ignite the plasma torch for at least 30 min before use, to allow stabilization.

Measure the emission intensity using the ICP-atomic emission spectrometer using the internal standard method.

When using devices for a computer system preparation of a working curve, standardization (drift correction, normalization, recalibration) and measurement of iron concentration shall be in accordance with the operating manual for the device’s computer system software.

7.5.2 Preparation of working curve

Spray a portion of the calibration solutions into the argon plasma of an ICP-atomic emission spectrometer, measure the emission intensity of iron and the internal standard elements (Co, Y, La, or Ti) simultaneously at the wavelength shown in Table 1.

Table 1 — Element and measurement wavelength

Element	**Wavelength** nm
Fe	238,20 or 259,94
Co	228,62
Y	371,03
La	398,85
Ti	255,60 or 333,21[a]

[a] For Fe 238,20 nm line, the Ti 255,59 nm line should used as the internal standard line 9 and for the Fe 259,94 nm, the Ti 333,211 nm line should be used the as internal standard line.

Calculate the intensity ratios of the emission intensities of iron and the internal standard elements using Equation (1).

$$R = I_{\mathrm{Fe}}/I_{\mathrm{IS}} \tag{1}$$

where

R is the intensity ratio of iron and the internal standard element;

I_{Fe} is the emission intensity of iron;

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

Prepare the relation curve between the obtained intensity ratios and the iron concentration, in mg/100 ml. Ensure that the graph achieves better than 0,999 correlation and is suitably linear.

Prepare the working curve for a sample by parallel shifting of the relation curve, so as to let the curve pass through the origin of the coordinate axes.

7.5.3 Measurement of iron concentration

Spray a portion of the test solutions into the argon plasma of an ICP-atomic emission spectrometer.

Measure the emission intensity of iron and the internal standard elements (Co, Y, La, or Ti) simultaneously at the wavelength shown in Table 1. Calculate the intensity ratios of the emission intensities of iron and the internal standard element using Equation (1).

Determine the iron concentration (mg/100 ml) of the test solutions from the working curve.

8 Expression of result

The mass fraction of iron in the test portion, w_{Fe}, expressed as a percentage, is given by the Equation (2).

$$w_{\mathrm{Fe}} = \frac{\rho_1 - (\rho_2 - \rho_3)}{m} \times 100 \times 10^{-3} \qquad (2)$$

where

ρ_1 is the concentration of iron in the test solution, expressed in mg/100 ml;

ρ_2 is the concentration of iron in the blank solution, expressed in mg/100 ml;

ρ_3 is the concentration of iron in the titanium (4.8) used in 7.3.2 or 7.3.3, expressed in mg/100 ml;

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

Calculate the mass fraction of iron in the test portion to the third decimal place.

The difference in results of two parallel determinations of the mass fraction of iron in the same sample shall not be greater than the tolerance [Equation (3), (4), (5), (6), (7) or (8)]. If the difference exceeds the tolerance, the analysis shall be repeated.