INTERNATIONAL STANDARD

ISO 22960

First edition
2008-01-15

Titanium and titanium alloys — Determination of iron — Molecular absorption spectrometry using 1, 10-phenanthroline

Titane et alliages de titane — Dosage du fer — Spectrométrie d'absorption moléculaire par la 1,10-phénanthroline

Reference number
ISO 22960:2008(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.

COPYRIGHT PROTECTED DOCUMENT

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 22960 was prepared by Technical Committee ISO/TC 79, *Light metals and their alloys*, Subcommittee SC 11, *Titanium*.

Titanium and titanium alloys — Determination of iron — Molecular absorption spectrometry using 1, 10-phenanthroline

1 Scope

This International Standard specifies a molecular absorption spectrometric method using 1, 10-phenanthroline 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 0,005 % to 2,0 %.

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 hydrochloric acid and hydrofluoric acid. Add nitric acid to oxidize titanium. Then add boric acid, tartaric acid, ammonium acetate and hydroxyl ammonium chloride. Add 1, 10-phenanthroline to make a 1,10-phenanthroline-iron complex. Determine the iron concentration in the test portion using a spectrophotometer.

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.

4.3 Nitric acid (1+1)

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

4.4 Hydrofluoric acid (1+1)

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

4.5 Boric acid

4.6 Tartaric acid solution (200 g/l)

4.7 Ammonium acetate solution (500 g/l)

4.8 Hydroxyl ammonium chloride solution (100 g/l)

4.9 1, 10-phenanthroline solution (100 g/l)

Dissolve 2,4 g of 1,10-phenanthrolinium chloride monohydrate in 1 000 ml of water, or dissolve 2,0 g of 1,10-phenanthroline monohydrate in 100 ml of ethanol [minimum purity 95 % (volume fraction)] and dilute to 1 000 ml with water.

4.10 Iron standard solution

4.10.1 Iron standard stock 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 (4.3) to oxidize iron and heat to evaporate nitrogen oxides. After cooling, transfer to a 1 000 ml volumetric flask, make up to the mark with water and mix.

4.10.2 Iron standard solution (0,050 mg Fe/ml)

Pipette 10,00 ml of iron standard stock solution (4.10.1) to a 100 ml volumetric flask, make up to the mark with water and mix. Prepare this solution on the day of use.

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 Analytical balance, sensitive to 0,1 mg.

5.4 Spectrophotometer.

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 from the test sample as specified in Table 1 and weigh to the nearest 0,1 mg.

Table 1 — Recommended test portion masses and aliquot volume taken in 7.4

Mass fraction of iron %	Mass of test portion g	Volume of aliquot taken in 7.4 c) ml
⩾ 0,005 < 0,1	1,0	20,0
⩾ 0,1 < 0,4	1,0	10,0
⩾ 0,4 < 0,8	0,50	10,0
⩾ 0,8 ⩽ 2,0	0,20	10,0

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 working curve

Pipette 0 ml, 2 ml, 4 ml, 6 ml and 8 ml of iron standard solution (4.10.2) into five 100 ml volumetric flasks.

Add 15 ml of tartaric acid solution (4.6), 25 ml of ammonium acetate solution (4.7) and 5 ml of hydroxyl ammonium chloride solution (4.8) to each volumetric flask. Dilute to about 80 ml with water and mix.

Add 10 ml of 1,10-phenanthroline solution (4.9) to each volumetric flask, make up to the mark with water and mix.

After allowing to stand for about 20 min at room temperature, transfer a part of the solution into an absorption cell (10 mm), and measure the absorbance at a wavelength of 510 nm using water as a contrast solution.

Prepare the curve showing the relation between the obtained absorbance and the iron concentration, in milligrams per 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 the origin of the coordinate axes.

7.4 Preparation of test solution for presentation to spectrophotometer

Carry out the following steps.

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

b) Add 10 ml of hydrochloric acid (4.2) and 5 ml of hydrofluoric acid (4.4), cover with a polyethylene watch glass and heat gently on a water bath to decompose the test portion. Add 3 ml of nitric acid (4.3) and continue heating until nitrogen oxides evaporate. Then add 3 g of boric acid (4.5), stir to dissolve the boric acid and cool to room temperature.

c) Transfer the solution into a 100 ml volumetric flask and make up to the mark with water. Pipette a test solution into two volumetric flasks (A and B) (specified volume in Table 1) according to the mass fractions of iron in the sample.

d) Add 15 ml of tartaric acid solution (4.6), 25 ml of ammonium acetate solution (4.7) and 5 ml of hydroxyl ammonium chloride solution (4.8) to the volumetric flasks A and B. Dilute to about 80 ml with water and mix.

e) Add 10 ml of 1,10-phenanthroline solution (4.9) to volumetric flask A, and make up to the mark with water. To the volumetric flask B, add water to make up to the mark and mix.

7.5 Determination of iron concentration

After allowing to stand for about 20 min at room temperature, transfer a part of the solution into an absorption cell (10 mm), and measure the absorbance at a wavelength of 510 nm using the solution in flask B as the contrast solution.

Determine the iron concentration of the test solution from the working curve (7.3).

8 Expression of results

The mass fraction of iron in the test portion, w_{Fe}, expressed as a percentage, is given by the following equation:

$$w_{Fe} = \frac{\rho_1 - \rho_2}{\frac{m \times V}{100}} \times 100 \times 10^{-3} \qquad (1)$$

where

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

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

V is the volume of aliquot solution, expressed in 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 (2), (3), or (4)]. If the difference exceeds the tolerance, the analysis shall be repeated.