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Titanium and titanium alloys — Determination of iron — Atomic absorption spectrometry

Titane et alliages de titane — Dosage du fer — Spectrométrie d'absorption atomique

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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

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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 22961 was prepared by Technical Committee ISO/TC 79, *Light metals and their alloys*, Subcommittee SC 11, *Titanium*.

Titanium and titanium alloys — Determination of iron — Atomic absorption spectrometry

1 Scope

This International Standard specifies an atomic absorption 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 0,005 % 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, iron and other elements. Then add boric acid, determine the iron concentration in the test portion using an atomic absorption 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.

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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 Titanium

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

4.7 Iron standard solution

4.7.1 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 5ml of nitric acid (4.3) to oxidize iron and heat until nitrogen oxides evaporates. After cooling, transfer to a 1 000 ml volumetric flask, make up to the mark with water and mix.

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

Pipette 10,00 ml of iron standard stock solution (4.7.1) 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 Atomic absorption spectrometer.

Instrumental conditions:

Flame: air/acetylene

Wavelength: 248,3 nm

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 or 7.3.3.

7.3.2 In the case of a mass fraction of iron less than 0,1 %

Weigh six samples of titanium (4.6), each of 0,50 g, and transfer to polyethylene beakers (200 ml).

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 are evaporated. Then add 3 g of boric acid (4.5), stir to dissolve the boric acid and cool to room temperature.

Transfer the solution into a 100 ml volumetric flask and add, from a burette or pipette, 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml of iron standard solution (4.7.2). Make up to the mark with water and mix.

7.3.3 In the case of a mass fraction of iron more than 0,1 %

Weigh 0,50 g of titanium (4.6) and transfer to a polyethylene beaker (200 ml).

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 titanium. Add 3 ml of nitric acid (4.3) and continue heating until nitrogen oxides are evaporated. Then add 3 g of boric acid (4.5), stir to dissolve the boric acid and cool to room temperature.

Transfer the solution into a 100 ml volumetric flask.

Pipette 20,0 ml or 5,0 ml of the solution according to the mass fraction of iron in the sample (see Table 1) into six volumetric flasks. Add 8 ml or 9,5 ml of hydrochloric acid (4.2) according to the mass fraction of iron in the sample (see Table 1). Add, from a burette or pipette, 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml of iron standard solution (4.7.2). Make up to the mark with water and mix.

7.4 Preparation of test solution for presentation to atomic absorption spectrometer

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

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 are evaporated. Then add 3 g of boric acid (4.5), stir to dissolve the boric acid and cool to room temperature.

Transfer the solution into a 100 ml volumetric flask, make up to the mark with water and mix.

In case the mass fraction of iron is more than 0,1 %, pipette 20,0 ml or 5,0 ml of the solution according to the mass fraction of iron in the sample into a 100 ml volumetric flask. Add 8 ml or 9,5 ml of hydrochloric acid (4.2) according to the aliquot volume, and make up to the mark with water (see Table 1).

Table 1 — Mass fractions of iron and aliquot volume

Mass fraction of iron %	**Aliquot volume** ml	**Added volume of HCl** (4.2) ml
$\geqslant 0,1 < 0,5$	20,0	8
$\geqslant 0,5 \leqslant 2$	5,0	9,5

7.5 Determination of iron concentration

7.5.1 General

The instrument shall be set up with an iron hollow cathode lamp in accordance with the manufacturer's recommendation. The wavelength shall be set to 248,3 nm. An oxidizing acetylene-air flame is used.

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 air and acetylene flame of an atomic absorption spectrometer of which the zero point has been regulated by using water, measure the absorbance at a wavelength of 248,3 nm, prepare the relation curve between the obtained absorbance and iron concentration in milligrams per 100 ml.

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 air and acetylene flame of an atomic absorption spectrometer of which the zero point has been regulated by using water, measure the absorbance at a wavelength of 248,3 nm, and determine the iron concentration (mg/100 ml) of the test solutions from the working curve.

8 Expression of results

8.1 In the case of a mass fraction of iron less than 0,1 % (m/m)

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

$$W_{\text{Fe}} = \frac{\rho_1 - (\rho_2 - \rho_3)}{m} \times 100 \times 10^{-3} \qquad (1)$$

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 (4.6) used in 7.3.2 or 7.3.3, expressed in mg/100ml;

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 oxygen content in the same sample shall not be greater than the tolerance [Equation (3), (4) or (5)]. If the difference exceeds the tolerance, the analysis shall be repeated.

8.2 In the case of a mass fraction of iron more than 0,1 %

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

$$W_{\text{Fe}} = \frac{\rho_4 - \left[\rho_5 - \left(\rho_3 \times \frac{V}{100}\right)\right]}{m} \times 100 \times 10^{-3} \qquad (2)$$

where

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

ρ_4 is the concentration of iron in the aliquot test solution expressed in mg/100 ml;

ρ_5 is the concentration of iron in the aliquot blank solution expressed in mg/100 ml;

V is the volume of aliquot, 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 (3), (4) or (5)]. If the difference exceeds the tolerance, the analysis shall be repeated.