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

Warning

This document is not an ISO International Standard. It is distributed for review and comment. It is subject to change without notice and may not be referred to as an International Standard.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.
Copyright notice

This ISO document is a working draft or committee draft and is copyright-protected by ISO. While the reproduction of working drafts or committee drafts in any form for use by participants in the ISO standards development process is permitted without prior permission from ISO, neither this document nor any extract from it may be reproduced, stored or transmitted in any form for any other purpose without prior written permission from ISO.

Requests for permission to reproduce this document for the purpose of selling it should be addressed as shown below or to ISO’s member body in the country of the requester:

Japanese Industrial Standards Committee (JISC):
c/o Technical Regulation, Standards and Conformity Assessment Policy Unit
Ministry of International Trade and Industry
1-3-1, Kasumigaseki, Chiyoda-ku Tokyo 100
TEL: + 81 3 35 01 94 71
FAX: + 81 3 35 80 86 37
E-mail: isojisc@mext.go.jp

as appropriate, of the Copyright Manager of the ISO member body responsible for the secretariat of the TC or SC within the framework of which the draft has been prepared]

Reproduction for sales purposes may be subject to royalty payments or a licensing agreement.

Violators may be prosecuted.
## Contents

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Foreword</td>
<td>iv</td>
</tr>
<tr>
<td>Introduction</td>
<td>v</td>
</tr>
<tr>
<td>1 Scope</td>
<td>1</td>
</tr>
<tr>
<td>2 Normative references</td>
<td>1</td>
</tr>
<tr>
<td>3 Principle</td>
<td>1</td>
</tr>
<tr>
<td>4 Reagents</td>
<td>1</td>
</tr>
<tr>
<td>4.1 General</td>
<td>1</td>
</tr>
<tr>
<td>4.2 Hydrochloric acid (1+1)</td>
<td>2</td>
</tr>
<tr>
<td>4.3 Nitric acid (1+1)</td>
<td>2</td>
</tr>
<tr>
<td>4.4 Hydrofluoric acid (1+1)</td>
<td>2</td>
</tr>
<tr>
<td>4.5 Boric acid</td>
<td>2</td>
</tr>
<tr>
<td>4.6 Titanium</td>
<td>2</td>
</tr>
<tr>
<td>4.7 Iron standard solution</td>
<td>2</td>
</tr>
<tr>
<td>4.7.1 Iron standard stock solution (0, 500 mg Fe/ml)</td>
<td>2</td>
</tr>
<tr>
<td>4.7.2 Iron standard solution (0.050mgFe/ml)</td>
<td>2</td>
</tr>
<tr>
<td>5 Apparatus</td>
<td>2</td>
</tr>
<tr>
<td>5.1 General</td>
<td>2</td>
</tr>
<tr>
<td>5.2 Volumetric glassware</td>
<td>2</td>
</tr>
<tr>
<td>5.3 Analytical balance</td>
<td>2</td>
</tr>
<tr>
<td>5.4 Atomic absorption spectrometer</td>
<td>3</td>
</tr>
<tr>
<td>6 Sample</td>
<td>3</td>
</tr>
<tr>
<td>6.1 Sampling</td>
<td>3</td>
</tr>
<tr>
<td>6.2 Test portion</td>
<td>3</td>
</tr>
<tr>
<td>7 Procedure</td>
<td>3</td>
</tr>
<tr>
<td>7.1 Number of determination</td>
<td>3</td>
</tr>
<tr>
<td>7.2 Blank test</td>
<td>3</td>
</tr>
<tr>
<td>7.3 Preparation of calibration solution</td>
<td>3</td>
</tr>
<tr>
<td>7.3.1 General</td>
<td>3</td>
</tr>
<tr>
<td>7.3.2 In case of iron content less than 0, 1 % (m/m)</td>
<td>3</td>
</tr>
<tr>
<td>7.3.3 In case of iron content more than 0,1 % (m/m)</td>
<td>3</td>
</tr>
<tr>
<td>7.4 Preparation of test solution for presentation to atomic absorption spectrometer</td>
<td>4</td>
</tr>
<tr>
<td>7.5 Determination of iron concentration</td>
<td>4</td>
</tr>
<tr>
<td>7.5.1 General</td>
<td>4</td>
</tr>
<tr>
<td>7.5.2 Preparation of working curve</td>
<td>4</td>
</tr>
<tr>
<td>7.5.3 Measurement of iron concentration</td>
<td>5</td>
</tr>
<tr>
<td>8 Expression of result</td>
<td>5</td>
</tr>
<tr>
<td>8.1 In case of iron content less than 0, 1%(m/m)</td>
<td>5</td>
</tr>
<tr>
<td>8.2 In case of iron content more than 0, 1 % (m/m)</td>
<td>5</td>
</tr>
<tr>
<td>9 Precision</td>
<td>6</td>
</tr>
</tbody>
</table>
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 Electro-technical Commission (IEC) on all matters of electro-technical 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/CD 22961 was prepared by Technical Committee ISO/TC 79, Light metals and their alloys, Subcommittee SC 11, Titanium.
Introduction
Titanium and titanium alloys — Determination of iron — Atomic absorption spectrometry

1 Scope

This International Standard specifies atomic absorption spectrometry for determination of the iron content in titanium and titanium alloys.

The method is applicable to titanium and titanium alloys with iron content in the range from 0,005 % (m/m) to 2,0 % (m/m)

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
ISO 648:1977, Laboratory glassware -- One-mark pipettes
ISO 1042:1983, Laboratory glassware — one-mark volumetric flasks
ISO 3696: 1987, Water for analytical laboratory use - Specifications and test methods
ISO 4787: 1984, Laboratory glassware

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 iron concentration in the test portion using 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.
4.2 Hydrochloric acid (1+1)

Add slowly 500 ml of hydrochloric acid \((\rho_{20} 1.16 \text{ g/ml to 1.19 g/ml})\) to 500 ml of water.

4.3 Nitric acid (1+1)

Add slowly 500 ml of nitric acid \((\rho_{20} 1.42 \text{ g/ml})\) to 500 ml of water.

4.4 Hydrofluoric acid (1+1)

Add carefully and slowly 100 ml of hydrofluoric acid \((\rho_{20} 1.14 \text{ g/ml})\) to 100 ml of water.

4.5 Boric acid

4.6 Titanium

Minimum 99% (m/m) purity and the iron content as low as possible, and already known. If iron content is unknown, the content should be determined by the future ISO standard now being discussed (ISO/CD22960).

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 % (m/m)], 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 evaporated. 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 (0.050mgFe/ml)

Pipette 10,00ml of iron standard stock solution (4.7.1) to a 100ml 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

Class A complying with ISO 384, ISO 648 and ISO 1042, and 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 determination

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 case of iron content less than 0,1 % (m/m)

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

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

(3) Transfer the solution into a 100 ml volumetric flask and add from 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 case of iron content more than 0,1 % (m/m)

(1) Weigh 0,50 g of titanium (4.6) and transfer to a polyethylene beaker (200 ml).
(2) 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 evaporated. Then add 3 g of boric acid (4.5) and stir to dissolve the boric acid and cool to room temperature.

(3) Transfer the solution into a 100 ml volumetric flask.

(4) Pipette 20,0 ml or 5,0 ml of the solution according to the iron contents 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 iron contents in the sample (see table 1). Add from 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

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

(2) 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 3ml of nitric acid (4.3) and continue heating until nitrogen oxides evaporated. Then add 3 g of boric acid (4.5) and stir to dissolve the boric acid and cool to room temperature.

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

NOTE In case of iron content is more than 0,1 % (m/m), pipette 20,0 ml or 5,0 ml of the solution according to the iron contents in the sample into 100 ml volumetric flask. Add 8 ml or 9,5 ml of hydrochloric acid (4.2) according to the aliquot volume, make up to the mark with water (see table 1).

Table 1 — Iron contents and aliquot volume

<table>
<thead>
<tr>
<th>Iron content % (m/m)</th>
<th>Aliquot volume (ml)</th>
<th>Added volume of HCl (4.2) (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>≥ 0,1 &lt; 0,5</td>
<td>20,0</td>
<td>8</td>
</tr>
<tr>
<td>≥ 0,5 &lt; 2 &lt; 0,1</td>
<td>5,0</td>
<td>9,5</td>
</tr>
</tbody>
</table>

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.

NOTE When using devices with computer system preparation of a working curve, standardization (drift correction, normalization, recalibration) and measuring of iron concentration shall be in accordance with the operating manual for the device 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 248,3 nm in wavelength, prepare the relation curve between the obtained absorbance and iron concentration in milligram per 100 ml.
Prepare the working curve for a sample by parallel shifting the relation curve so as to let the curve pass 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 248.3 nm in wavelength, and determine the iron concentration (mg/100ml) of the test solutions from the working curve.

8 Expression of result

8.1 In case of iron content less than 0.1% (m/m)

The iron content of the test portion \( W_{Fe} \), expressed as a percentage by mass, is given by the following equation:

\[
W_{Fe} = \frac{A1 - (A2 - A3)}{m} \times 100 \times 10^{-3}
\]

where

\( W_{Fe} \) is the iron content of test portion expressed in percent by mass;

\( A1 \) is the concentration of iron in the test solution expressed in mg/100 ml;

\( A2 \) is the concentration of iron in the blank solution expressed in mg/100 ml;

\( A3 \) is the concentration of iron in the blank solution expressed in mg/100 ml;

\( m \) is the concentration of iron in the used titanium (4.5) in 7.3.1 or 7.3.2 expressed in milligram.

Calculate the iron content of the test portion to the third decimal place.

NOTE 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 case of iron content more than 0.1 % (m/m)

The iron content of the test portion \( W_{Fe} \), expressed as a percentage by mass, is given by the following equation:

\[
W_{Fe} = \frac{A4 - \left[ A5 - \left( A3 \times \frac{B}{100} \right) \right]}{m} \times 100 \times 10^{-3}
\]

where

\( W_{Fe} \) is iron content of test portion expressed in percent by mass;

\( A3 \) is the concentration of iron in the used titanium in 7.3.1 or 7.3.2 expressed in milligram;

\( A4 \) is the concentration of iron in the aliquot test solution expressed in mg/100ml;

\( A5 \) is the concentration of iron in the aliquot blank solution expressed in mg/100ml;
B is volume of aliquot expressed in ml;

m is the mass of test portion expressed in gram.

Calculate the iron content of the test portion to the third decimal place.

NOTE The difference in results of two parallel determinations of iron 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.

9 Precision

The tolerance of this analytical method is expressed by the following equations;

Repeatability limit

\[ 2.8 \times [0.010 \times (\text{Fe content } \%) + 0.0004] \% (m/m) \] (3)

Within-laboratory-reproducibility limit

\[ 2.8 \times [0.0149 \times (\text{Fe content } \%) + 0.0004] \% (m/m) \] (4)

Between-laboratory-reproducibility limit

\[ 2.8 \times [0.0270 \times (\text{Fe content } \%) + 0.0023] \% (m/m) \] (5)