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Titanium and titanium alloys — Determination of oxygen — Infrared method after fusion under inert gas

Titane et alliages de titane — Dosage de l'oxygène — Méthode par infrarouge après fusion sous gaz inerte

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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 22963 was prepared by Technical Committee ISO/TC 79, *Light metal and their alloys*, Subcommittee SC 11, *Titanium*.

Titanium and titanium alloys — Determination of oxygen — Infrared method after fusion under inert gas

1 Scope

This International Standard specifies an infrared method after fusion under inert gas for the determination of the mass fraction of oxygen in titanium and titanium alloys.

The method is applicable to titanium and titanium alloys with a mass fraction of oxygen in the range from 0,02 % to 0,4 %.

2 Principle

The test portion, plus flux (platinum or nickel metal) is fused in a graphite crucible under an inert gas stream (He or Ar) using an impulse furnace. The released oxygen combines with carbon to form carbon monoxide. Depending on the instrument design, the carbon monoxide is oxidized into carbon dioxide or left as carbon monoxide and swept by the inert gas stream into an infrared detector.

Infrared absorption of the carbon monoxide or dioxide is measured, against a calibration curve made of titanium standard sample.

3 Reagents

3.1 General

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

3.2 **Magnesium perchlorate**, Mg(ClO4) 2 (commercial designation: anhydrone)

This is used in the instrument to absorb water. Use the purity specified by the instrument manufacturer.

3.3 **Sodium hydroxide on clay** (commercial designation: ascarite)

Used in some instruments to absorb carbon dioxide. Use purity specified by the instrument manufacturer.

3.4 Copper oxide

Used in some instruments to oxidize carbon monoxide to carbon dioxide. Use the purity specified by the instrument manufacturer.

3.5 Helium or argon

Use the purity and type (He or Ar) specified by the instrument manufacturer.

3.6 Nickel cleaning solution

Mix 75 ml of acetic acid, 25 ml of nitric acid and 2 ml of hydrochloric acid.

3.7 Titanium cleaning solution

3.7.1 Titanium cleaning solution (A)

Mix 100 ml of nitric acid (1+1) and 5 ml of hydrofluoric acid.

3.7.2 Titanium cleaning solution (B)

Mix 90 ml of nitric acid (1+1) and 30 ml of hydrofluoric acid.

3.8 Acetone

3.9 Nickel

Capsule, wire or baskets of minimum purity 99 % (mass fraction), with a mass fraction of oxygen less than 0,005 %. Nickel is immersed in the warm nickel cleaning solution (3.6) for 30 s. Then rinse with water, ethanol, and acetone, each for 30 s, and store the nickel in acetone until used. Just before use, it should be dried with air.

3.10 Platinum

Sheet (thickness $<$ 1 mm), wire (ø $<$ 3 mm) or foil (thickness $<$ from 0,01 to 0,03 mm) of minimum purity 99 % (mass fraction) and the mass fraction of oxygen is less than 0,005 %. Platinum is washed with acetone and stored in a desiccator until used.

3.11 Titanium standard sample

Select only titanium and titanium alloy standards. Select one containing approximately from 0,2 % to 0,35 % (mass fraction) oxygen. The accuracy of the test method is largely dependent upon the oxygen values assigned to the reference materials and upon the homogeneity of these materials. Thus, the titanium standard sample should be used with standard reference materials or certified reference materials.

4 Apparatus

4.1 Instrument.

The instrument required for fusion of the test portion under an inert gas stream (He or Ar), using an impulse furnace and measurement of the carbon monoxide or dioxide extracted, may be obtained commercially from a number of manufacturers.

4.2 Graphite crucible, use high-purity crucibles suitable for the instrument.

4.3 Glass-wool filters.

4.4 Tweezer, made of solvent- and acid-resistant plastic (used during the sample preparation process).

5 Sample

5.1 Sampling

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

5.2 Preparation of test portions

The optimum test portion is a pin (approximately 5 mm in diameter and nominally weighing from 0,06 g to 0,14 g) or a cube (of sides approximately 3 mm and nominally weighing from 0,06 g to 0,14 g).

Cut off a test portion (from 0,08 g to 0,14 g) from the laboratory sample. The test portion shall be cut off by machining, using no lubricating fluid and avoiding test-portion over-heating.

Clean the sample surface in accordance with method a), b) or c).

a) **Method a)** Leach the test portion in the titanium cleaning solution (3.7.1) at about 20 °C until the surface is clean. This will normally require approximately 50 s. Immediately remove the reacting test portion with tweezers and rinse it twice with water and once with ethanol and acetone, and allow to air dry. This test portion should now weigh between 0,05 g and 0,13 g. The test portion should be reserved in a desiccator.

b) **Method b)** Leach the test portion in the titanium cleaning solution (3.7.2) for 5 s from the start of a violent reaction of the test portion with the solution. Immediately remove the reacting test portion with tweezers and rinse it twice with water and once with ethanol and acetone, and allow to air dry. This test portion should now weigh between 0,05 g and 0,13 g. The test portion should be reserved in a desiccator.

c) **Method c)** File or cut off all outside edges, retaining only fresh surfaces, and finishing by rinsing with acetone and air drying. This test portion should now weigh between 0,05 g and 0,13 g.

Weigh the test portion to the nearest 0,1 mg.

6 Procedure

6.1 Number of determinations

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

6.2 Preparation of instrument

Assemble the apparatus as recommended by the manufacturer. Make the required power, gas and water connections.

Place a graphite crucible (4.2) on the furnace pedestal and then degas by heating above the degassing temperature of the sample.

Continuously heat at the oxygen-extraction temperature and record the analyzer reading signal. Repeat this operation until the stable value of the analyzer reading signal is obtained.

Check that the glass-wool filters (4.3) are clean and change them as often as necessary.

If the electricity supply has been switched off for a long time, allow the instrument to stabilize for the time recommended by manufacturer.

After changing the filter (4.3) and/or reagents (3.2, 3.3 and 3.4), or when the apparatus has been inoperative for a period, stabilize the instrument by carrying out trial analyses, the results of which are to be disregarded. Then proceed with blank, calibration and preparation tests as indicated in 6.3, 6.4, and 6.5 before analyzing the sample.

NOTE The appropriate degas temperature of the sample and oxygen extraction temperature are determined by the manufacturer's instruction.

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

6.3 Blank test

Prior to the determination, carry out the following blank test in duplicate.

Proceed as directed in 6.5 with the graphite crucible, and analyze the nickel or platinum (same kind of metal and same mass during sample analysis) without the test portion.

Obtain the reading of the blank test.

The mean value of the blank test must be sufficiently low when compared to the content to be determined in the test portion.

If the blank values are abnormally high, investigate and eliminate the source of contamination.

If the mean value of the blank is satisfactory, it may then be introduced into the blank subtraction device of the instrument.

6.4 Calibration

Prior to the determination, carry out the following calibration.

Proceed as directed in 6.5, using a titanium standard sample (3.11) instead of the test portion.

Subtract the mean of the blank value (6.3) from the analyzer reading signal.

Repeat the process above several times.

Calculate the intermediate oxygen equivalence factor (f) of the analyzer reading signal and calculate the oxygen equivalence factor (F) using Equations (1) and (2),

$$f_i = m_{\mathrm{O}}/S_i \tag{1}$$

$$I = l \sim n$$

$$F = \frac{\sum\limits_{i=1}^{n} f_i}{n} \tag{2}$$

where

F is the oxygen equivalence factor, expressed in mg, of the oxygen analyzer reading signal;

f is the intermediate oxygen equivalence factor, expressed in mg, of the oxygen analyzer reading signal;

m_{O} is the mass of oxygen in the weighted titanium standard sample, expressed in mg (calculated in Equation (3);

S is the intermediate analyzer reading signal after subtracting the mean value of the blank.

NOTE 1 Calculate the mass of oxygen in the weighted titanium standard sample as follows:

$$m_{\mathrm{O}} = \left[(G \times P)/100 \right] \times 10^{3} \tag{3}$$

where

m_{O} is the mass of oxygen in the weighted titanium standard sample, expressed in mg;

G is the mass of titanium standard sample, expressed in g;

P is the mass fraction of oxygen in the titanium standard sample, expressed in percent.

NOTE 2 Refer to the manufacturer’s instructions for proper blanking procedures.

6.5 Procedure

Assemble the apparatus, calibrate it, and test the performance as directed in 6.2, 6.3 and 6.4.

Clean the electrode with a brush and vacuum cleaner before each determination.

Wrap a test portion (5.2) in nickel (3.9) or platinum (3.10) and place it in the sample drop-port.

NOTE The mass of nickel or platinum must exceed the mass of the test portion by at least a factor of ten.

Place a graphite crucible (4.2) on the furnace pedestal and then degas by heating above the degassing temperature of the sample.

Place the test portion in nickel (3.9) or platinum (3.10) in the degassed crucible, entering a test portion weight.

Operate the furnace in accordance with the manufacturer’s instructions for fusion, extraction of oxygen and measurement of an infrared absorption.

At the end of the fusion and measuring cycle, remove and discard the crucible, and record the analyzer reading signal.

During the sample analysis run, the analysis of a titanium standard sample at regular intervals is recommended for monitoring drift when validating the initial calibration.

6.6 Calculation

The mass fraction of oxygen in the test portion, W_{O}, expressed in a percentage, is calculated by Equation (4).

$$W_{\mathrm{O}} = \left[\left(A_1 - A_2 \right) \times F \times 100/m \right] \times 10^{-3} \tag{4}$$

where

A_1 is the analyzer reading signal of the sample analysis;

A_2 is the mean value of the analyzer reading signal of the blank test;

F is the oxygen equivalence factor, expressed in mg, of the oxygen/analyzer reading signal;

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

NOTE The most modern fusion equipment gives the results directly in percent of oxygen, so that post-analysis calculations are normally not required.

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

7 Precision

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

Within-laboratory-repeatability limit

$$2{,}8 \times [0{,}004\,8 \times (W_{\mathrm{O}}) + 0{,}001\,5]\ \%\ \text{(mass fraction)} \tag{5}$$

Within-laboratory-reproducibility limit

$$2{,}8 \times [0{,}014 \times (W_{\mathrm{O}}) + 0{,}000\,5]\ \%\ \text{(mass fraction)} \tag{6}$$

Between-laboratory-reproducibility limit

$$2{,}8 \times [0{,}017\,1 \times (W_{\mathrm{O}}) + 0{,}001]\ \%\ \text{(mass fraction)} \tag{7}$$

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