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Steel and iron — Determination of oxygen — Infrared method after fusion under inert gas

Acier et fonte — Dosage de l'oxygène — Méthode par infrarouge après fusion sous gaz inerte

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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 17053 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

Steel and iron — 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 oxygen in steel and iron.

The method is applicable to mass fractions of oxygen between 0,000 75 % and 0,01 %.

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 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

ISO 14284:1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Principle

Fusion of a test portion in a single-use graphite crucible under helium gas at a minimum temperature of 2 000 °C. Combination of the oxygen from the sample with carbon from the crucible to form carbon monoxide. Eventually, transformation of carbon monoxide into carbon dioxide.

Measurement of infrared absorption of the carbon monoxide or dioxide, against a calibration curve made of potassium nitrate.

4 Reagents and materials

4.1 General

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

4.2 Helium, of high purity, total impurity content 0,000 5 % (mass fraction).

An oxidation catalyst [copper(II) oxide or platinum] tube heated to a temperature above 450 °C shall be used prior to a purifying unit, when the presence of organic contaminants is suspected in the helium.

4.3 Magnesium perchlorate, Mg $(ClO_4)_2$, (commercial designation: anhydrone), particle size from 1,2 mm to 2,0 mm, or anhydrous calcium sulfate, (commercial designation: drierite), particle size from 0,6 mm to 0,85 mm.

4.4 Sodium hydroxide, on granulated support, (commercial designation: ascarite), particule size from 0,7 mm to 1,2 mm.

4.5 Copper oxide, CuO.

4.6 Tin or nickel capsules, for example diameter 5 mm, length 13 mm, mass 160 mg, with low mass fraction of oxygen, less than 0,1 µg/mg of oxygen.

Cut off the upper part of the tin capsule to reduce the mass to 50 mg or the length to 8 mm in order to remove the contaminated part.

4.7 Water of high purity, deionised, prepared just before use.

4.8 Potassium nitrate, KNO_3, of high purity, maximum total impurity content 0,000 5 % by mass.

Dry before use at 100°C to 105°C for 2 h, and allow to cool in a dessicator.

4.9 Potassium nitrate stock solution, corresponding to 4 mg/ml of oxygen.

Weigh, to the nearest 0,1 mg, 8,416 g of potassium nitrate (4.8).

Place in a 100 ml beaker and dissolve in about 50 ml of water (4.7).

Transfer quantitatively to a 1 000 ml volumetric flask, dilute to the mark with water (4.7), and mix.

1 ml of the potassium nitrate stock solution contains 4 mg of oxygen as potassium nitrate.

4.10 Potassium nitrate diluted stock solution, corresponding to 0,4 mg/ml of oxygen.

This solution must be prepared freshly before use.

Transfer 10 ml of the potassium nitrate stock solution to a 100 ml volumetric flask, dilute to the mark with water (4.7), and mix.

1 ml of the potassium nitrate stock solution contains 0,4 mg of oxygen as potassium nitrate.

4.11 Potassium nitrate standard solutions

These solutions must be prepared freshly before use.

Transfer the volume listed in Table 1 of the potassium nitrate stock solution (4.9) and of the potassium nitrate diluted stock solution (4.10) to a series of nine 100 ml volumetric flasks, dilute to the mark with water (4.7), and mix.

100 µl of each of the potassium nitrate standard solutions contain the mass of oxygen (as potassium nitrate) listed in the last column of Table 1.

4.12 Graphite powder, of high purity, with low oxygen content.

Table 1 — Potassium nitrate standard solution

Potassium nitrate standard solution	Volume of potassium nitrate stock solution (4.9) added (except for solution 4.11.2)	Mass of oxygen in 1 ml of the potassium nitrate standard solution	Mass of oxygen in 100 μl of the potassium nitrate standard solution
4.11.1	0 ml [a]	0 mg	0 μg
4.11.2	3 ml of potassium nitrate diluted stock solution (4.10)	0,012 mg	1,2 μg
4.11.3	1,0 ml	0,04 mg	4 μg
4.11.4	2,0 ml	0,08 mg	8 μg
4.11.5	3,0 ml	0,12 mg	12 μg
4.11.6	5,0 ml	0,20 mg	20 μg
4.11.7	10,0 ml	0,40 mg	40 μg
4.11.8	20,0 ml	0,80 mg	80 μg
4.11.9	30,0 ml	1,20 mg	120 μg

[a] Zero member: The standard solution (4.11.1) is in fact the water (4.7) used for preparing the solutions.

5 Apparatus

5.1 General

The apparatus required for fusion of the test portion, and measurement of the carbon monoxide or dioxide extracted, may be obtained commercially from a number of manufacturers. Follow the manufacturer's instructions for the operation of the instrument.

5.2 Graphite crucible, single-use.

Use high-purity crucibles suited to the apparatus.

5.3 Crucible tongs, for handling the crucibles used.

5.4 Glass-wool filters.

5.5 Micropipette, 100 μl, the limit of error shall be less than 1 μl.

6 Sampling

Carry out sampling in accordance with ISO 14284 or appropriate national standards for steel.

7 Procedure

SAFETY INSTRUCTIONS — The risk involved when using an apparatus for fusing the test portion are mainly risks of burns. It is therefore necessary to use crucible tongs (5.3) and appropriate containers for the used crucibles.

7.1 General instructions

Check that glass-wool filters (5.4) 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 the manufacturer.

After changing the filters (5.4) and/or reagents (4.3, 4.4 and 4.5), 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 7.3, 7.4 and 7.5, before analysing the sample.

If the instrument used provides a direct reading in percentage of oxygen, adjust the instrument reading for each calibration range as follows.

Read the content of a certified reference material (CRM) of high oxygen content at various steps of heating power. The required heating power for the determination of test samples is that at which the reading levels off.

In order to determine a high-alloy test sample, a high-alloy CRM shall be used to know the required heating power.

7.2 Test portion

Prepare the test portion of approximately 1 g in accordance with ISO 14284 or appropriate national standards for steel.

Weigh the test portion to the nearest 1 mg.

7.3 Blank test

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

Proceed as in 7.6 without the test portion, entering a mass of 1 g, and setting the blank subtraction of the instrument to zero.

Obtain the reading of the blank test.

The mean value of the blank test shall be sufficiently low when compared to the mass fraction of oxygen to be determined in the test portion.

For a mean value below 8 µg/g of oxygen, the mean blank value and the difference between the two blank values shall both not exceed 0,5 µg/g of oxygen. For a mean value above 8 µg/g of oxygen, the mean blank value and the difference between the two blank values shall both not exceed 2 µg/g of oxygen.

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 in the blank subtraction device of the instrument.

7.4 Calibration

Prior to the determination, carry out the following calibration.

Prepare, according to 4.11, at least five solutions chosen among solutions 4.11.1 to 4.11.9 indicated in Table 1, according to the level of mass fraction of oxygen to be determined.

Using the micropipette (5.5), transfer 100 µl of each of the freshly prepared potassium nitrate standard solutions (4.11.1 to 4.11.9) to each tin or nickel capsule (4.6), respectively, and dry at 90 °C to 95 °C for 2 h.

The drying of the calibration solutions in the tin or nickel capsules shall be done stepwise, in order to avoid splashing and loss of calibration material due to excessive heating before complete vaporisation of the water.

Gently press the tin or nickel capsule and proceed as in 7.6, using the tin or nickel capsule as a test portion, entering a sample mass of one gram, and setting the sample fusion temperature to 2 000 °C to limit tin vaporization.

Prepare a calibration graph by plotting the reading obtained from the instrument against micrograms of oxygen from the last column of Table 1.

7.5 Preparation test

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

Choose a CRM or a working sample to be prepared before oxygen analysis, suited to the expected mass fraction of oxygen in the samples to be analysed.

Proceed as in 7.2 for the preparation of this preparation test sample.

Proceed as in 7.6 for the determination on the preparation test sample.

The results shall be within the limits of a control chart for the preparation test sample.

If not, check the preparation conditions, and rerun the sample preparation test.

7.6 Determination

7.6.1 Method 1

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

Place a graphite crucible (5.2) in the furnace and then degas by heating above 2 100 °C to 2 800 °C.

Put the test portion with an empty tin or nickel capsule (4.6) into the degassed crucible, entering a sample mass of 1 g

Operate the furnace in accordance with the manufacturer's instructions.

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

7.6.2 Method 2

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

Place a graphite crucible (5.2) in the furnace and then degas by heating above 2 100 °C to 2 800 °C. Repeat degassing once if possible with the apparatus used.

NOTE The addition of 0,3 g of graphite powder (4.12) before degassing can lead to a faster reduction of surface contamination and therefore give a better separation of the two surface contamination and bulk oxygen peaks.

After a cooling period of 5 s to 10 s, put the test portion with an empty tin or nickel capsule (4.6) into the degassed crucible, entering a sample mass of one gram.

Select a suitable heating rate to separate a first peak corresponding to surface contamination (signal 1), and a second peak corresponding to the bulk oxygen (signal 2), operating the furnace in accordance with the manufacturer's instructions.

The separation of surface contamination and bulk oxygen can be achieved by a two-stage fusion, with parameters which can depend on the equipment used in accordance with laboratory experience and the manufacturer's instructions, for example:

- an increase of the furnace temperature with a heating rate of less than 1 °C/s by a dial reading, until a first peak corresponding to surface contamination appears:
- followed by a rapid increase of the furnace temperature to 2 000 °C to 2 800 °C, giving a second peak corresponding to bulk oxygen evolution.

At the end of the fusion and measuring cycle, remove and discard the crucible, and record the analyser reading signal 2 (bulk oxygen).

8 Expression of results

8.1 Method of calculation

Convert the analyser reading (total signal of method 1, or signal 2 bulk oxygen of method 2) of the test portion to micrograms of oxygen (m_0), by means of the calibration graph (see 7.4).

The mass fraction of oxygen, expressed as a percentage w_O (expressed in micrograms per gram) is given by the equation:

$$w_\mathrm{O} = \frac{m_0}{m}$$

where

m_0 is the mass, expressed in micrograms, of oxygen in the test portion;

m is the mass, in grams, of the test portion (see 7.2).

The result is the mean value of at least two determinations made on the same sample.

The individual values may be given in order to provide additional information.

For a mean value below a mass fraction of 0,000 8 % of oxygen, the difference between two individual determinations shall not exceed a mass fraction of 0,000 2 % of oxygen.

For a mean value below a mass fraction of 0,005 0 % of oxygen, the difference between two individual determinations shall not exceed a mass fraction of 0,000 5 % of oxygen.

For a mean value above a mass fraction of 0,005 0 % of oxygen, the difference between two individual determinations shall not exceed 10 % of the mean value.

If the 10 % limit is exceeded, perform additional determinations sufficient to eliminate variation due to sample preparation. Then report the number of remaining values and their range in accordance with 9 d).

The decision of discarding an outlying result is more efficiently taken when a chart recorder is fitted to the apparatus.

For example, a faulty preparation of the sample surface will produce an important peak at the beginning of the fusion.

8.2 Precision

A planned trial of this method was carried out by 15 laboratories, at 9 levels of oxygen, each laboratory making three determinations of mass fraction of oxygen at each level.

NOTE 1 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725-1; i.e. one operator, same apparatus, identical operating conditions, same calibration, and a minimum period of time.

NOTE 2 The third determination was carried out at a different time (on a different day), by the same operator as in Note 1, using the same apparatus with a new calibration.

The test samples used and mean results obtained are listed in Table A.1.

The results obtained were treated statistically in accordance with ISO 5725-1, ISO 5725-2 and ISO 5725-3.

The data obtained showed a logarithmic relationship between mass fraction of oxygen and repeatability limit (r) and reproducibility limits (Rw and R) of the test results (see Note 3) as summarized in Table 2. The graphical representation of the figures is given in Annex B.

NOTE 3 From the two values obtained on day 1, the repeatability limit (r) and reproducibility limit (R) were calculated using the procedure specified in ISO 5725-2. From the first value obtained on day 1 and the value obtained on day 2, the within-laboratory reproducibility limit (Rw) was calculated using the procedure specified in ISO 5725-3.

Table 2 — Results for repeatability limit and reproducibility limits

Mass fraction of oxygen	**Repeatability limit**	**Reproducibility limits**	
%	r	Rw	R
0,000 75	0,000 25	0,000 39	0,000 46
0,001	0,000 28	0,000 43	0,000 57
0,002	0,000 40	0,000 57	0,000 95
0,003	0,000 49	0,000 67	0,001 29
0,005	0,000 64	0,000 82	0,001 89
0,01	0,000 90	0,001 08	0,003 18

9 Test report

The test report shall include the following information:

a) all information necessary for the identification of the sample, the laboratory and the date of analysis,

b) the method used by reference to this International Standard,

c) the results and the form in which they are expressed,

d) any unusual features noted during the determination,

e) any operation not specified in this International Standard, or any operation which may have influenced the results.