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Chemical analysis of ferrous materials - Determination of oxygen content in steel and iron - Part 2: Infrared method after fusion under inert gas

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Chemische Analyse von Eisenwerkstoffen - Bestimmung des Sauerstoffgehalts von Stahl und Eisen - Teil 2: Messung der Infrarotabsorption nach Aufschmelzen unter Inertgas

Analyse chimique des matériaux ferreux - Détermination de la teneur en oxygene de l'acier et de la fonte - Partie 2: Méthode par absorption dans l'infrarouge apres fusion sous gaz inerte

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This document EN 10276-2:2003 has been prepared by Technical Committee ECISS/TC 20 “Methods of chemical analysis of ferrous products”, the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2004, and conflicting national standards shall be withdrawn at the latest by January 2004.

This document includes a Bibliography.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies an infrared method after fusion under inert gas for the determination of oxygen in steel and iron.

The method is applicable to oxygen contents between 0,0005 % (m/m) and 0,01 % (m/m).

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 10276-1	*Chemical analysis of ferrous materials – Determination of oxygen in steel and iron – Part 1: Sampling and preparation of steel samples for oxygen determination.*
EN ISO 14284	*Steel and iron – Sampling and preparation of samples for the determination of chemical composition (ISO 14284:1996).*

3 Principle

Fusion of a test portion in a single-use graphite crucible under helium gas at a minimum temperature of 2000° 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 and use of a calibration curve plotted using the measurements obtained with potassium nitrate.

4 Reagents and materials

During the analysis, unless otherwise stated, only reagents of recognised analytical grade shall be used. The following reagents and materials shall be used:

4.1 Helium, high purity, total impurity content 0,0005 % (m/m).

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.

4.2 Magnesium perchlorate, Mg $(ClO_4)_2$, (commercial designation: anhydrone), particle size from 1,2 mm to 2,0 mm, or **Anhydrous Calcium sulphate, ($CaSO_4$)**, (commercial designation: drierite), particle size from 0,6 mm to 0,85 mm.

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

4.4 Copper oxide, CuO

4.5 Platinum, Pt

4.6 Tin or nickel capsules, For example ∅ 5 mm, length 13 mm, weight 160 mg, with low oxygen content, less than 0,1 µg/mg of oxygen.

Cut off the upper part of the tin capsule to reduce the weight to 50 mg or the length to 8 mm.

4.7 Water, high purity, deionized, prepared just before use.

4.8 Potassium nitrate, KNO3, high purity, maximum total impurity content 0,0005 % (m/m).

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 1000 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 shall be prepared freshly before use.

Transfer 10 ml of the potassium nitrate stock solution in 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 shall 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) in 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.

Table 1 — Potassium nitrate standard solutions

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.

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

5 Apparatus

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. The manufacturer's instructions for the operation of the instrument shall be followed.

5.1 Graphite crucible, single-use

High purity crucibles suited to the apparatus shall be used.

5.2 Crucible tongs shall be used for handling the crucibles.

5.3 Glass wool filters

5.4 Micropipette, 100 µl, shall be used, limit of error shall be less than 1 µl.

6 Sampling

Sampling shall be carried out in accordance with EN ISO 14284 and EN 10276-1.

7 Procedure

7.1 General

The risk involved when using an apparatus for fusing the test portion are mainly risks of burns. Crucible tongs (5.2) and appropriate containers for the used crucibles shall be used.

7.2 General instructions

It shall be checked that glass wool filters (5.3) are clean and they shall be changed as often as necessary.

If the electricity supply has been switched off for a long time, the instrument shall be allowed to stabilise for the time recommended by the manufacturer.

After changing the filters (5.3) and/or reagents (4.2, 4.3 and 4.4), or when the apparatus has been inoperative for a period, the instrument shall be allowed to stabilise by carrying out trial analyses, the result of which are to be disregarded, then proceed with blank calibration and preparation tests as indicated in 7.4, 7.5 and 7.6 before analysing the sample.

If the instrument used provides a direct reading in percentage of oxygen, the instrument reading shall be adjusted for each calibration range as follows:

Using a CRM with a high oxygen content, record the contents measured at different levels of furnace 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.

7.3 Test portion

Prepare the test portion of approximately 1 g in accordance with EN 10276-1.

Weigh the test portion to the nearest 1 mg.

7.4 Blank test

Prior to the determination, the following blank test shall be carried out in duplicate.

Proceed as in 7.7 without test portion, entering a weight 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 oxygen content to be determined in the test portion.

For mean values below 8 µg/g of oxygen, neither the mean blank value nor the difference between the two blank values should exceed 0,5 µg/g of oxygen. For mean values above 8 µg/g of oxygen, neither the mean blank value nor the difference between the two blank values should exceed 2 µg/g of oxygen.

If the blank values are abnormally high, the source of contamination shall be investigated and eliminated.

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