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Unalloyed steel — Determination of low carbon content —

Part 2:
Infrared absorption method after combustion in an induction furnace (with preheating)

Acier non allié — Détermination des faibles teneurs en carbone —

Partie 2: Méthode par absorption dans l'infrarouge après combustion dans un four à induction (avec préchauffage)

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 15349-2:1999), which has been technically revised in order to re-assess the precision data.

A list of all parts in the ISO 15349 series can be found on the ISO website.

Unalloyed steel — Determination of low carbon content —

Part 2: Infrared absorption method after combustion in an induction furnace (with preheating)

1 Scope

This document specifies an infrared absorption method after combustion in an induction furnace for the determination of the low carbon content in unalloyed steel.

The method is applicable to carbon mass fraction contents between 0,000 3 % and 0,009 % .

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Pre-heating of a test portion at low temperature followed by its combustion in presence of an accelerator at a high temperature in an induction furnace in a current of pure oxygen.

Transformation of carbon into carbon dioxide and/or carbon monoxide.

Measurement by infrared absorption of the carbon dioxide and/or carbon monoxide evolved from steel and carried by a current of pure oxygen.

The calibration is carried out using sucrose or calcium carbonate.

5 Reagent

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only water with a low content of organic matter, i.e. grade 2 water as specified in ISO 3696.

5.1 Water, free from carbon dioxide

Boil water for 30 min, cool to room temperature and allow oxygen (5.2) or a high purity inert gas to bubble through it for 15 min. Prepare just before use.

5.2 Oxygen, minimum purity 99,95 % (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 in the oxygen is suspected.

The pressure of oxygen in the furnace is controlled by a pressure regulator designed especially for this purpose.

5.3 Pure iron, containing less than 0,000 3 % mass fraction of carbon or having very low and known carbon content.

5.4 Solvent, appropriate for cleaning greasy or dirty test samples, for example, acetone

5.5 Accelerator, copper plate (see NOTE 1) or pellet type tin and granular tungsten mixture (see NOTE 2) containing less than 0,000 3 % mass fraction of carbon or having very low and known carbon content.

NOTE 1 Plate shape or granular copper (about 0,1 g/plate) should be used after the following treatment. Heat the copper plate at 450 °C to 600 °C for 10 min in a current of oxygen or air and cool in a desiccator without grease. This treatment shall be carried out just before use.

NOTE 2 Pellet type tin (about 0,2 g/pellet) and granular tungsten, 12 mesh to 20 mesh should be used after the following treatment. Heat the tungsten at 450 °C in air for 10 min and cool in a desiccator without grease. Clean the tin for more than 5 min by use of hydrochloric acid in an ultrasonic cleaner, rinse with water and dry in air. These treatments shall be carried out just before use.

5.6 Sucrose, standard solutions

Weigh, to the nearest 0,1 mg, the seven masses of sucrose ($C_{12}H_{22}O_{11}$) (analytical standard grade) indicated in Table 1, previously dried at 100 °C to 105 °C for 2,5 h and cooled in a desiccator, and transfer to seven 100 ml beakers.

Add 30 ml of water (5.1) to dissolve, transfer to seven 100 ml one-mark volumetric flasks quantitatively, dilute to the mark with water (5.1) and mix.

Table 1 — Standard solution series of sucrose

Standard solution reference number	Mass of sucrose g	Corresponding mass of added carbon µg	Carbon content in 1 g of the test portion % mass fraction
1	0 [a]	0	0
2	0,010 0	4,21	0,000 42
3	0,025 0	10,53	0,001 05
4	0,060 0	25,26	0,002 53
5	0,120 0	50,53	0,005 05
6	0,180 0	75,79	0,007 58

[a] Zero member

Table 1 *(continued)*

Standard solution reference number	Mass of sucrose g	Corresponding mass of added carbon µg	Carbon content in 1 g of the test portion % mass fraction
7	0,240 0	101,1	0,010 11
[a] Zero member			

5.7 Calcium carbonate

Dry calcium carbonate [minimum purity: 99,9 % (mass fraction)] at 180 °C for 1 h and cool in a desiccator before use.

5.8 Magnesium perchlorate, anhydrous, [$Mg(ClO4)_2$], particle size: from 0,7 mm to 1,2 mm

5.9 Inert ceramic (attapulgus clay) impregnated with sodium hydroxide, particle size: from 0,6 mm to 1,2 mm

6 Apparatus

The apparatus required for combustion in an induction furnace and the subsequent infrared absorption measurement of the evolved carbon dioxide and/or carbon monoxide may be obtained commercially from a number of manufacturers. Follow the manufacturer's instructions for the operation of the instrument.

Common features of such systems are described in annex A.

All laboratory glassware shall be class A, in accordance with ISO 648 or ISO 1042 as appropriate.

Common features of such systems are discussed in annex A.

Ordinary laboratory apparatus and the following should be used.

6.1 Ceramic crucible, capable of withstanding combustion in an induction furnace

Just before use, ignite crucibles in an electric furnace in a current of oxygen or air for not less than 2 h at more than 1 200 °C and keep in a desiccator or closed container before use.

NOTE For the determination of low carbon contents it is advisable to ignite crucibles at 1 350 °C in a current of oxygen.

6.2 Tin capsule, about 6 mm in diameter, 18 mm in height, 0,3 g in mass and approximately 0,4 ml in volume of known and very low carbon content less than 0,000 3 % mass fraction of carbon or having very low and known carbon content.

Tin capsules should be used after the following treatment.

Rinse the capsule in hydrochloric acid (ρ approximately = 1,19 g/ml, diluted 1+1) for 5 min while shaking frequently, wash it thoroughly with water and dry. Store it in a clean glass bottle.

6.3 Glass-fiber filter, 21 mm in diameter

Glass-fiber filters should be used after the following treatment.

Heat the glass-fiber filter at 500 °C to 550 °C for 30 min and more in air and cool in a desiccator without grease. Store it in a clean glass bottle.

6.4 Micropipette, 100 µl, limit of error should be less than 1 µl.

6.5 Microbalance, weighing to the nearest 0,1 µg.

6.6 Muffle or wire-wound furnace, regulated at 400 °C to 500 °C.

7 Sampling and preparation of the test samples

Sampling and preparation of the samples shall be carried out in accordance with ISO 14284 or appropriate national standards. The chip size of test sample should be between 0,75 mm and 2,0 mm.

8 Procedure

SAFETY INSTRUCTIONS The risks related to combustion analysis are mainly burns in pre-igniting the ceramic crucible and in the combustion. Use crucible tongs at all times and suitable containers for the used crucibles. Normal precautions for handling oxygen cylinders shall be taken. Oxygen from the combustion process shall be removed effectively from the apparatus since a high concentration of oxygen in a confined space can present a fire hazard.

8.1 General

Purify the oxygen supply using tubes packed with the inert ceramic (attapulgus clay) impregnated with sodium hydroxide (5.9) and magnesium perchlorate (5.8), and maintain a quiescent flow rate whilst on standby.

Maintain a glass-fiber filter or a stainless-steel filter screen as a dust collector between the furnace chamber and the analyser. Clean and charge as necessary. The furnace chamber, pedestal post and filter trap should be cleaned frequently to remove oxide build-up.

The oxygen flow rate may vary from one instrument to another, but is usually about 2,0 l/min during the combustion period, according to the nature of the material. The temperature reached during the combustion stage depends on the power of the HF generator, the geometry of the furnace chamber, the induction coil and the type and quantity of sample in the crucible. This temperature may be in the order of 1 700°C, or more.

When the main supply is switched on after being out of action for any length of time, allow the equipment to stabilize for the time recommended by the equipment manufacturers for the stabilization of equipment.

After cleaning the furnace chamber and/or changing filters or after the equipment has been inoperative for a period, stabilize the apparatus by burning several samples, of similar type to the samples to be analysed, prior to setting up for analysis.

Flush oxygen through the apparatus and adjust the instrument controls to give a zero reading.

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

Select a certified reference material with a carbon content close to the maximum carbon content in the calibration series, and measure the carbon content of the certified reference material in the manner specified in 8.4.

Adjust the reading of the instrument to the certified value.

NOTE This adjustment shall be made before the calibration as specified in 8.5. It cannot replace or correct the calibration.

8.2 Test portion

If necessary, degrease the test sample by cleaning in a suitable solvent (5.4). Evaporate the last traces of the solvent by heating cautiously.

Weigh, to the nearest 0,1 mg, approximately 1,0 g of the test sample.

8.3 Blank test

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

Weigh, to the nearest 0,1 mg, approximately 1,0 g of the pure iron (5.3) and transfer to a ceramic crucible (6.1).

Place the ceramic crucible containing the pure iron in the muffle or wire-wound furnace (6.6) heated to 420 °C ± 10 °C for about 5 min to 10 min.

Remove the ceramic crucible containing the pure iron from the muffle or wire-wound furnace and immediately add the appropriate mass of the accelerator (5.5) (see NOTE 1) and one tin capsule (6.2) (see NOTES 2 and 3) or one glass-fiber filter (6.3) (see NOTE 4) into the ceramic crucible containing the pure iron.

Treat the crucible and contents as specified in 8.4.2.

Obtain the reading of the blank tests and convert it to micrograms, to the nearest 0,1 µg, of carbon by means of the calibration curve (see 8.5).

The blank value is obtained by subtracting the mass of carbon in the pure iron (5.3) used from the blank tests mean value.

The mean blank value ($m_{C,0}$) is calculated from the two blank values to the nearest 0,1 µg.

The mean blank value shall not exceed 3,0 µg of carbon, and the difference between the two blank values shall not exceed 2,0 µg of carbon. If these values are abnormally high, investigate and eliminate the source of contamination.

NOTE 1 The quantity of accelerators will depend on the individual characteristics of the instrument and the type of material being analysed. The amount used shall be sufficient for complete combustion.

NOTE 2 In cases where the calibration curve of 8.5.1 is applied, a tin capsule prepared as follows may be used. Using the micropipette (6.4), transfer 100 µl of water (5.1) to a tin capsule (6.2) and dry at 90 °C for 2 h.

NOTE 3 In cases where the tin capsule (6.2) is used, after transferring it to the ceramic crucible (6.1), it may be pressed lightly against the bottom of the crucible.

NOTE 4 In cases where the calibration curve of 8.5.1 is applied, a glass-fiber filter prepared as follows may be used. Using the micropipette (6.4), transfer 100 µl of water (5.1) to a glass-fiber filter (6.3) and dry at 90 °C for 2 h.

8.4 Determination

8.4.1 Pre-treatment of the test portion

Place the test portion (8.2) into a ceramic crucible (6.1).

Place the ceramic crucible containing the test portion in the muffle or wire-wound furnace (6.6) heated to 420 °C ± 10 °C for about 5 min to 10 min.

Remove the ceramic crucible containing the test portion from the muffle or wire-wound furnace and immediately add the appropriate mass of the accelerator (5.5) (see NOTE 1 in 8.3) and, when sucrose is used as calibration reagent, one tin capsule (6.2) (see NOTES 2 and 3 in 8.3) or one glass-fiber filter (6.3) (see NOTE 4 in 8.3) into the ceramic crucible containing the test portion.

8.4.2 Combustion of the test portion

Immediately, place the ceramic crucible and contents on the pedestal post, raise to the combustion position and lock the system. Operate the furnace in accordance with the manufacturer's instructions.