

ISO/TC 17/SC 1

Secretariat: JISC

Voting begins on:
2021-04-15

Voting terminates on:
2021-06-10

Unalloyed steel — Determination of low carbon content —

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

*Aciers non alliés — Détermination des faibles teneurs en carbone —
Partie 2: Méthode par absorption dans l'infrarouge après combustion
dans un four à induction (avec préchauffage)*

<https://standards.iteh.ai/catalog/standards/sist/9f9029f4-d55c-41a8-a54b-c6d54848b593/iso-fdis-15349-2>

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.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

ISO/CEN PARALLEL PROCESSING



Reference number
ISO/FDIS 15349-2:2021(E)

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO/FDIS 15349-2
<https://standards.iteh.ai/catalog/standards/sist/9f9029f4-d55c-41a8-a54b-c6d54848b593/iso-fdis-15349-2>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2021

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Reagents	2
6 Apparatus	3
7 Sampling and preparation of the test samples	4
8 Procedure	4
8.1 General	4
8.2 Test portion	5
8.3 Blank test	5
8.4 Determination	5
8.4.1 Pre-treatment of the test portion	5
8.4.2 Combustion of the test portion	6
8.5 Establishment of the calibration curve	6
8.5.1 Calibration with sucrose standard solutions	6
8.5.2 Calibration with calcium carbonate	6
9 Expression of results	7
9.1 Method of calculation	7
9.2 Precision	7
10 Test report	8
Annex A (informative) Features of commercial induction furnaces and infrared carbon analysers	9
Annex B (informative) Additional information on the international inter-laboratory test	11
Annex C (informative) Graphical representation of precision data	12
Bibliography	13

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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 459/SC 2, *Methods of chemical analysis for iron and steel*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 15349-2:1999), which has been technically revised. The main changes compared to the previous edition are as follows:

- normative references have been revised;
- the precision data has been updated;
- the former Table B.3 has been deleted;
- the text has been improved editorially.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 contents between 0,000 3 % (mass fraction) and 0,009 % (mass fraction).

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 sample and carried by a current of pure oxygen.

The calibration is carried out using sucrose or calcium carbonate.

5 Reagents

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 bubble oxygen (5.2) or a high purity inert gas through it for 15 min. Prepare just before use.

5.2 Oxygen, minimum purity 99,95 % (volume fraction).

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

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 a very low and known carbon content.

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

5.5 Accelerator, common accelerators are copper, tungsten-tin mixture, iron, tungsten or nickel. Copper, tungsten-tin mixture or tungsten containing less than 0,000 3 % carbon (mass fraction) may be used.

Copper plate or pellet type tin and granular tungsten mixture containing less than 0,000 3 % (mass fraction) of carbon or having a very low and known carbon content.

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.

If necessary, wash three times with acetone by decantation to remove organic contaminants and dry at room temperature.

Pellet type tin (about 0,2 g/pellet) and granular tungsten, 1,68 mm (12 mesh) to 0,853 mm (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 with 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 masses of sucrose ($C_{12}H_{22}O_{11}$) indicated in Table 1, previously dried at 100 °C to 105 °C for 2,5 h and cooled in a desiccator, and transfer to a series of seven 100 ml beakers.

Add 30 ml of water (5.1) to dissolve, transfer quantitatively into a series of 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	Carbon content in 1 g of the test portion
		µg	% mass fraction
1	0 ^a	0	0
2	0,010 0	4,21	0,000 42

^a Zero member.

Table 1 (continued)

Standard solution reference number	Mass of sucrose g	Corresponding mass of added carbon	Carbon content in 1 g of the test portion
		µg	% mass fraction
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
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 2,0 mm.

Phosphorous pentoxide may also be used by some instrument manufacturers.

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

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.

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 the 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.

For the determination of low carbon contents, it is advisable to ignite the 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-fibre filter, 21 mm in diameter.

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

Heat the glass-fibre 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 size of the 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 crucibles 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

iTeh STANDARD PREVIEW

Purify the oxygen supply by means of tubes packed with the inert ceramic (attapulugus clay) impregnated with sodium hydroxide (5.9) and magnesium perchlorate (5.8), and maintain a quiescent flow rate whilst on standby. Other purifying reagents are recommended by some manufacturers of carbon analysers.

<https://standards.iteh.ai/catalog/standards/sist/9f9029f4-d55c-41a8-a54b-c6d54848b593/iso-fdis-15349-2>

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

The oxygen flow rate can 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 high frequency 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 switched off for any period of time, allow the equipment to stabilize for the time recommended by the manufacturer of the equipment 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.

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 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 $420\text{ °C} \pm 10\text{ °C}$ 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) and one tin capsule (6.2) or one glass-fibre filter (6.3) into this ceramic crucible.

Treat the crucible and contents as specified in 8.4.2.

For the blank test, in the cases where the calibration curve specified in 8.5.1 is used, transfer into the tin capsule (6.2) 100 µl of water (5.1), by means of the micropipette (6.4), and dry at 90° C for 2 h.

For the blank test, in the cases where the calibration curve specified in 8.5.2 is used, transfer 100 µl of water (5.1) to a glass-fiber filter (6.3), by means of the micropipette (6.4), and dry at 90° C for 2 h.

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.

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.

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.

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 $420\text{ °C} \pm 10\text{ °C}$ 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 8.3) and, when sucrose is used as

calibration reagent, one tin capsule (6.2) or one glass-fibre filter (6.3) (see 8.3) into this 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.

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

8.5 Establishment of the calibration curve

8.5.1 Calibration with sucrose standard solutions

8.5.1.1 Preparation of the calibration series

Using a micropipette (6.4), transfer 100 µl, to the nearest 1 µl, of each of the sucrose standard solutions (5.6) into a series of seven tin capsules (6.2) (see 8.3) or glass-fibre filters (6.3) and dry at 90 °C for 2 h.

Cool to room temperature in a desiccator.

The weight of 100 µl sucrose standard solution may be confirmed by weighing to the nearest 1 mg, if necessary.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

8.5.1.2 Measurements

Weigh, to the nearest 0,1 mg, approximately 1,0 g of 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 5 min to 10 min.

Remove the ceramic crucible containing the pure iron from the muffle or wire-wound furnace.

Immediately transfer the tin capsule (6.2) or glass-fibre filter (6.3) containing sucrose to the ceramic crucible containing the pure iron, and cover with the same quantity of the accelerator (5.5) (see 8.3) as that used for the test portion.

Treat the crucible and contents as described in 8.4.2.

8.5.1.3 Plotting the calibration curve

Obtain the net readings by subtracting the reading of the zero member from that of each member of the calibration series.

Prepare the calibration curve by plotting the net readings against the mass of carbon of each member of the calibration series expressed in micrograms to the nearest 0,1 µg.

8.5.2 Calibration with calcium carbonate

8.5.2.1 Preparation of the calibration series

Using a microbalance (6.5), weigh, to the nearest 0,1 µg, the masses of calcium carbonate (5.7) indicated in Table 2 and transfer to a series of ceramic crucibles (6.1).

8.5.2.2 Measurements

Perform as described in 8.5.1.2 but a tin capsule or glass-filter is not necessary.