
**Unalloyed steel — Determination of
low carbon content —**

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

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)*



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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).

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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 the 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 the 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, 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
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, $[\text{Mg}(\text{ClO}_4)_2]$, 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.

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 or 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

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.

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 1700 °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 $420\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ 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 $90\text{ }^{\circ}\text{C}$ 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.

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 $420\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ 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).

Table 2 — Calibration series prepared with calcium carbonate

Mass of calcium carbonate (5.7) µg	Corresponding mass of carbon added µg	Carbon content in 1 g of the test portion % mass fraction
0 ^a	0	0
40	4,8	0,000 48
85	10,2	0,001 02
210	25,2	0,002 52
420	50,4	0,005 04
630	75,6	0,007 56
850	102,0	0,010 20

^a Zero member.

8.5.2.2 Measurements

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

8.5.2.3 Plotting the calibration curve

Perform as described in 8.5.1.3.

9 Expression of results

9.1 Method of calculation

Convert the analyser reading of the test portion to micrograms of carbon by means of the calibration curve (see 8.5).

The carbon content, expressed as a percentage by mass, w_c , is given by Formula (1):

$$w_c = \frac{(m_{c,1} - m_{c,0})}{m \times 10^6} \times 100 \quad (1)$$

where

$m_{c,1}$ is the mass of carbon in the test portion, expressed in micrograms;

$m_{c,0}$ is the mass of carbon in the blank test (see 8.3), expressed in micrograms;

m is the mass of the test portion (see 8.2), expressed in grams.

9.2 Precision

A planned trial of this method was carried out by 19 laboratories, using 11 levels of carbon content, each laboratory making three determinations of the carbon content at each level (see NOTES 1 and 2).

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 composition of the test samples used is given in Table B.1 and the mean results obtained are given in Table 3.

The results obtained were treated statistically in accordance with ISO 5725-2:1994¹⁾ and ISO 5725-3:1994.

Since the coefficients of correlation of the relations between m and r , R_w and R are lower than 0,9 only the experimental precision data are presented (see Table 3).

Table 3 — Experimental precision data

Carbon content % mass fraction		Repeatability limit r % mass fraction	Reproducibility limits R_w and R % mass fraction	
Certified	Found			
0,000 247	0,000 35	0,000 13	0,000 14	0,000 38
0,000 5	0,000 48	0,000 10	0,000 19	0,000 30
0,000 5	0,000 54	0,000 16	0,000 15	0,000 32
0,000 7	0,000 51	0,000 21	0,000 23	0,000 37
0,001 2	0,001 18	0,000 18	0,000 29	0,000 55
0,001 9	0,002 00	0,000 23	0,000 28	0,000 70
0,002 4	0,002 56	0,000 19	0,000 58	0,001 03
0,003 7	0,004 01	0,000 39	0,000 51	0,000 79
0,005	0,004 87	0,000 13	0,000 39	0,000 64
0,005 9	0,006 30	0,000 27	0,000 50	0,000 98
0,008 3	0,008 84	0,000 27	0,000 42	0,000 84

10 Test report

The test report shall include the following information:

- All information necessary for the identification of the sample, the laboratory and the date of analysis or of the test report;
- Method used by reference to this document, i.e. ISO 15349-2:2021;
- Results and unit in which they are expressed;
- Any unusual features noted during the determination;
- Any operation not specified in this document, or any optional operation that might have influenced the results.

1) Withdrawn.

Annex A

(informative)

Features of commercial induction furnaces and infrared carbon analysers

A.1 Source of oxygen

The oxygen source is fitted with a fine regulating valve and a pressure gauge. A pressure regulator is required to control the oxygen pressure to the furnace according to the manufacturer's specification. This is usually 0,028 MPa.

A.2 Purifying unit

This unit contains inert ceramic impregnated with sodium hydroxide in a carbon dioxide absorbing tube, and magnesium perchlorate in a dehydration tube.

A.3 Flowmeter

The flowmeter is capable of measuring a flow of oxygen of 0 l/min up to 4 l/min.

A.4 Induction furnace

A.4.1 The combustion furnace consists of an induction coil and a high-frequency generator. The furnace chamber consists of a silica tube that fits inside the induction coil. This tube has metal plates at the top and bottom which are sealed to the tube by O-rings.

A.4.2 Gas inlet and outlet points are made through the metal plates.

A.4.3 The generator is usually a 1,5 kVA to 2,5 kVA apparent power unit, but the frequency used by certain manufacturers may be different. Values of 2 MHz to 6 MHz, 15 MHz, and 20 MHz are generally used. The power from the generator is fed to the induction coil which surrounds the silica furnace tube and is usually air-cooled.

A.4.4 The crucible containing the sample and accelerator is supported on a pedestal post which is precisely positioned so that, when raised, the metal in the crucible is correctly placed within the induction coil for effective coupling when the power is supplied.

A.4.5 The induction coil diameter, the number of turns, the furnace chamber geometry and the power of the generator determine the degree of coupling that can occur. These factors are determined by the instrument manufacturer.

A.4.6 The temperature attained during the combustion depends in part on the factors indicated in [A.4.5](#), but also on the characteristics of the sample in the crucible, the form of the test portion and the mass of materials. These factors may be varied to some extent by the operator.

It is essential that the oxides formed during combustion be removed by glass-fibre filtration, and the filter trap should be cleaned frequently to remove oxide build-up.

A.5 Dust collector

The dust collector is capable of collecting metal oxide dust in the current of oxygen from the furnace.

A.6 Desulfurization tube

This consists of a heated oxidation tube containing a platinum foil or platinized silica and a sulfur trioxide collector containing cellulose cotton.

A.7 Infrared gas analyser

A.7.1 In most instruments the gaseous products of combustion are transferred in a continuous flow of oxygen to the analyser system. The gases flow through an infrared cell, for example of the Luft type, where the absorption of the infrared radiation due to carbon dioxide and/or carbon monoxide is measured and integrated over a pre-programmed time period. The signal is amplified and converted to a digital display of the carbon content.

A.7.2 In some analysers, the products of combustion may be collected in oxygen in a fixed volume at controlled pressure and the mixture analysed for carbon dioxide and/or carbon monoxide.

A.7.3 Electronic controls are usually provided for adjusting the instrument zero, compensating for the blank, adjusting the slope of the calibration line and correcting for non-linear response. The analyser generally has a means of entering the mass of standard or test portion for automatic correction of the read-out.

A.7.4 Instruments may also be equipped with an integrated automatic balance for weighing the crucible, weighing the test portion and transferring the value of mass to the calculator.

In modern instruments, a microprocessor is included