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

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

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*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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15349 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

ISO 15349 consists of the following parts, under the general title *Unalloyed steel — Determination of low carbon content*:

- *Part 1: Infrared absorption method after combustion in an electric resistance furnace (by peak separation)*  
[Technical Report]
- *Part 2: Infrared absorption method after combustion in an induction furnace (with preheating)*  
[Technical Report]
- *Part 3: Infrared absorption method after combustion in an electric resistance furnace (with preheating)*  
[Technical Report]

Annexes A, B and C of this part of ISO 15349 are for information only.

# Unalloyed steel — Determination of low carbon content —

## Part 2:

## Infrared absorption method after combustion in an induction furnace (with preheating)

### 1 Scope

This part of ISO 15349 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 % (*m/m*) and 0,010 % (*m/m*).

### 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 15349. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 15349 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

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

Preheating of a test portion at low temperature and combustion of a test portion with 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 of infrared absorption of the carbon dioxide or carbon dioxide/carbon monoxide evolved from steel and carried by a current of pure oxygen.

Calibration graph is established using sucrose or calcium carbonate.

## 4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 3 water as specified in ISO 3696.

### 4.1 Water, free from carbon dioxide.

Boil water for 30 min, bubble with oxygen (4.2) while cooling to room temperature for 15 min. Prepare just before use.

### 4.2 Oxygen, 99,95 % (*m/m*) minimum.

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.

### 4.3 Pure iron, of known very low carbon content less than 0,000 3 % (*m/m*).

### 4.4 Suitable solvent, appropriate for washing greasy or dirty test samples, e.g., acetone.

### 4.5 Accelerator, copper plate (see NOTE 1) or pellet type tin and granular tungsten mixture (see NOTE 2) of known very low carbon content less than 0,000 3% (*m/m*).

NOTE 1 Copper plate (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 (only LECOCEL I is applicable) 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 in water and dry in air. These treatments shall be carried out just before use.

## 4.6 Standard substances

### 4.6.1 Sucrose, standard solution.

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

Add 30 ml of water (4.1) to dissolve, transfer to seven 100 ml one-mark volumetric flasks quantitatively, dilute to the mark with water (4.1) 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 % (m/m)
1	0 <sup>a</sup>	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

<sup>a</sup> Zero member.

#### 4.6.2 Calcium carbonate

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

**4.7 Magnesium perchlorate** [Mg(ClO<sub>4</sub>)<sub>2</sub>], particle size: from 0,7 mm to 1,2 mm.

**4.8 Inert ceramic (attapulugus clay) impregnated with sodium hydroxide**, particle size: from 0,7 mm to 1,2 mm.

## 5 Apparatus

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During the analysis, unless otherwise stated, use only ordinary laboratory apparatus.

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

The apparatus required for combustion in a high-frequency 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.

Features of commercial instruments are given in annex A.

**5.1 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.

**5.2 Tin capsule**, about 6 mm in diameter, 18 mm in height, 0,3 g in mass and approximately 0,4 ml in volume.

Tin capsules should be used after the following treatment.

Rinse the capsule in hydrochloric acid ( $\rho$  about 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.

**5.3 Glass-fiber filter**, 21 mm in diameter (for example, Whatman glass-fiber filter GF/F)

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.

- 5.4 Micropipette**, 100 µl, limit of error shall be less than 1 µl.
- 5.5 Microbalance**, weighing to the nearest 0,1 µg.
- 5.6 Muffle or wire-wound furnace**, regulated at 400 °C to 500 °C.

## 6 Sampling and preparation of the test samples

Sampling and preparation of the samples shall be carried out in accordance with ISO 14284. Prepare the chip size of test sample between 0,75 mm and 2,0 mm.

## 7 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.

### 7.1 General

Purify the oxygen supply using tubes packed with the inert ceramic (attapulugus clay) impregnated with sodium hydroxide (4.8) and magnesium perchlorate (4.7), and maintain a quiescent flow rate whilst on standby. Maintain a glass wool filter or a stainless steel net as a dust collector. Clean and charge as necessary. The furnace chamber, pedestal post and filter trap shall be cleaned frequently to remove oxide build-up.

Allow each item of equipment to stabilize for the time recommended by the equipment manufacturers when the main supply is switched on after being out of action for any length of time.

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 the 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 7.4.

Adjust the reading of the instrument to the certified value.

**NOTE** This adjustment shall be made before the calibration as specified in 7.5. It cannot replace or correct the calibration.

### 7.2 Test portion

Degrease the test sample by washing in a suitable solvent (4.4). Evaporate the last traces of the washing liquid by heating.

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

### 7.3 Blank test

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



Weigh, to the nearest 0,1 mg, approximately 1 g of the pure iron (4.3) and transfer to a ceramic crucible (5.1).

Place the ceramic crucible containing the pure iron in the muffle or wire-wound furnace (5.6) 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 and immediately add the appropriate mass of the accelerator(4.5) (see NOTE 1) and one tin capsule (5.2) (see NOTES 2 and 3) or one glass-fiber filter (5.3) (see NOTE 4) to the ceramic crucible containing the pure iron.

Treat the crucible and contents as specified in 7.4.2.

Obtain the reading of the blank tests and convert it to micrograms, to the nearest 0,1  $\mu\text{g}$ , of carbon by means of the calibration graph (see 7.5).

The blank value is obtained by subtracting the mass of carbon in the pure iron (4.3) used from the mass of carbon in the blank tests.

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

The mean blank value shall not exceed 3,0  $\mu\text{g}$  of carbon, and the difference between the two blank values shall not exceed 2,0  $\mu\text{g}$  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 graph of 7.5.1 is applied, a tin capsule prepared as follows may be used. Using the micropipette (5.4), transfer 100  $\mu\text{l}$  of water (4.1) to a tin capsule (5.2) and dry at  $90\text{ }^{\circ}\text{C}$  for 2 h.

NOTE 3 In cases where the tin capsule (5.2) is used, after transferring it to the ceramic crucible (5.1), it may be pressed lightly against the bottom of the crucible.

NOTE 4 In cases where the calibration graph of 7.5.1 is applied, a glass-fiber filter prepared as follows may be used. Using the micropipette (5.4), transfer 100  $\mu\text{l}$  of water (4.1) to a glass-fiber filter (5.3) and dry at  $90\text{ }^{\circ}\text{C}$  for 2 h.

## 7.4 Determination

### 7.4.1 Pre-treatment of the test portion

Transfer the test portion (see 7.2) to a ceramic crucible (5.1).

Place the ceramic crucible containing the test portion in the muffle or wire-wound furnace (5.6) 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 test portion from the muffle or wire-wound furnace and immediately add the appropriate mass of the accelerator (4.5) (see NOTE 1 in 7.3) and, when sucrose is used as calibration standard, one tin capsule (5.2) (see NOTES 2 and 3 in 7.3) or one glass-fiber filter (5.3) (see NOTE 4 in 7.3) to the ceramic crucible containing the test portion.

### 7.4.2 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.

## 7.5 Establishment of the calibration graph

### 7.5.1 In case of using the sucrose standard solution

#### 7.5.1.1 Preparation of the calibration series

Using a micropipette (5.4), introduce 100 µl, to the nearest 1 µl, of each of the sucrose standard solutions (4.6.1) into a series of seven tin capsules (5.2) (see NOTE 3 in 7.3) or glass-fiber filters (5.3) and dry at 90 °C for 2 h.

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

#### 7.5.1.2 Measurements

Weigh, to the nearest 0,1 mg, approximately 1,0 g of the pure iron (4.3) and transfer to a ceramic crucible (5.1).

Place the ceramic crucible containing the pure iron in the muffle or wire-wound furnace (5.6) 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 (5.2) or glass-fiber filter (5.3) containing sucrose to the ceramic crucible containing the pure iron, and cover with the same quantity of the accelerator (4.5) (see NOTE 1 in 7.3) as will be added to the test portion.

Treat the crucible and contents as described in 7.4.2.

#### 7.5.1.3 Plotting the calibration graph

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

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Prepare a calibration graph by plotting the net reading in micrograms, to the nearest 0,1 mg, of carbon for each member of the calibration series.

### 7.5.2 In case of using the calcium carbonate

#### 7.5.2.1 Preparation of the calibration series

Using a microbalance (5.5), weigh, to the nearest 0,1 µg, the seven masses of calcium carbonate (4.6.2) indicated in Table 2 and transfer to seven ceramic crucibles (5.1).

#### 7.5.2.2 Measurements

Perform as described in 7.5.1.2, but a tin capsule or glass-filter is not necessary.

Table 2 — Calibration series of calcium carbonate

Mass of calcium carbonate (4.6.2) µg	Corresponding mass of carbon added µg	Carbon content in 1 g of the test portion % (m/m)
0 <sup>a</sup>	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

<sup>a</sup> Zero member.

### 7.5.2.3 Plotting the calibration graph

Perform as described in 7.5.1.3.

## 8 Expression of results

### 8.1 Method of calculation

Convert the analyser reading of the test portion to micrograms of carbon by means of the calibration graph (see 7.5).

The carbon content, expressed as a percentage by mass,  $w_C$ , is given by the equation:

$$w_C = \frac{(m_{C,1} - m_{C,0})}{m \times 10^6} \times 100$$

$$= \frac{(m_{C,1} - m_{C,0})}{m \times 10^4}$$

where

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

$m_{C,0}$  is the mass, expressed in micrograms of carbon, in the blank test (see 7.3);

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

### 8.2 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 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.