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Steel and iron — Determination of total carbon content — Infrared absorption method after combustion in an induction furnace

Aciers et fontes — Détermination du carbone total — Méthode par absorption dans l'infrarouge après combustion dans un four à induction

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Foreword

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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 9556:1989), which has been technically revised.

The main changes are as follows:

- the normative references (Clause 2) has been revised;
- the mandatory terms and definitions clause (Clause 3) has been added, and subsequent clauses have been renumbered;
- precision data have been recalculated.

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.

Steel and iron — Determination of total carbon content — Infrared absorption method after combustion in an induction furnace

1 Scope

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

The method is applicable to carbon contents between 0,003 % (mass fraction) and 4,5 % (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 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 terminology databases for use in standardization at the following addresses:

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

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

4 Principle

Combustion of a test portion in presence of an accelerator at a high temperature in a high-frequency 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 carried by a current of oxygen.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 **Water**, free from carbon dioxide.

Boil water for 30 min, cool to room temperature and bubble with oxygen (5.2) for 15 min. Prepare just before use.

5.2 Oxygen, 99,5 % (volume fraction) 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 (see Annex C), when the presence of organic contaminants is suspected in the oxygen.

5.3 Pure iron, of known low carbon content less than 0,001 0 % (mass fraction).

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

5.5 Magnesium perchlorate [$Mg(ClO_4)_2$], particle size: from 0,7 mm to 1,2 mm.

5.6 Barium carbonate

Dry barium carbonate (minimum purity: 99,5 %) at 105 °C to 110 °C for 3 h and cool in a desiccator before use.

5.7 Sodium carbonate

Dry anhydrous sodium carbonate (minimum purity: 99,9 %) at 285 °C for 2 h and cool in a desiccator before use.

5.8 Accelerator: copper, tungsten-tin mixture or tungsten of known low carbon content less than 0,001 0 % (mass fraction).

5.9 Sucrose, standard solution, corresponding to 25 g of carbon per litre.

Weigh, to the nearest 1 mg, 14,843 g of sucrose ($C_{12}H_{22}O_{11}$) previously dried at 100 °C to 105 °C for 2,5 h and cooled in a desiccator.

Dissolve in about 100 ml of water (5.1), transfer quantitatively into a 250 ml one-mark volumetric flask, dilute to the mark with water (5.1) and mix.

1 ml of this standard solution contains 25 mg of carbon.

5.10 Sodium carbonate, standard solution, corresponding to 25 g of carbon per litre.

Weigh, to the nearest 1 mg, 55,152 g of sodium carbonate (5.7), dissolve in about 200 ml of water (5.1), transfer quantitatively into a 250 ml one-mark volumetric flask, dilute to the mark with water (5.1) and mix.

1 ml of this standard solution contains 25 mg of carbon.

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

6 Apparatus

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

All volumetric 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. Follow the manufacturer's instructions for the operation of the instrument.

Features of commercial instruments are given in Annex C.

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

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 low carbon content less than 0,001 0 % (mass fraction).

6.3 Ceramic crucible, capable of withstanding combustion in an induction furnace.

Ignite crucibles in an electric furnace in air or in a current of oxygen for not less than 2 h at 1 100 °C and store in a desiccator before use.

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

7 Sampling and sample preparation

The sampling and the sample preparation shall be carried out in accordance with ISO 14284 or appropriate national standards for steel and iron.

8 Procedure

WARNING — The risks related to combustion analysis are mainly burns in pre-igniting the ceramic crucibles and in the fusions. 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 operating instructions

Purify the oxygen supply using tubes packed with the inert ceramic (attapulgus clay) impregnated with sodium hydroxide (5.11) and magnesium perchlorate (5.5), 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 change as necessary. The furnace chamber, pedestal post and filter trap shall be cleaned frequently to remove oxide build-up.

Allow each item of the equipment to stabilize for the time recommended by the manufacturers of the equipment 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 a certified reference material with a carbon content close to the maximum carbon content in the calibration range, 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 carried out before the calibration specified in 8.5. It cannot replace or correct the calibration.

8.2 Test portion

Degrease the test sample by cleaning in an appropriate solvent (5.4). Evaporate the last traces of the solvent by heating.

Weigh, to the nearest 1 mg, approximately 1 g of the test sample for carbon contents less than 1,0 % (mass fraction) and approximately 0,5 g for carbon contents higher than 1,0 % (mass fraction).

NOTE The mass of the test portion can depend on the type of instrument used.

8.3 Blank test

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

8.3.2 Transfer a tin capsule (6.2) into a ceramic crucible (6.3). In cases where the calibration graphs of 8.5.1 or 8.5.2 are applied, prepare the tin capsule as follows: using the micropipette (6.1), transfer 100 µl of water (5.1) to the tin capsule (6.2) and dry at 90 °C for 2 h.

8.3.3 Press the tin capsule lightly against the bottom of the crucible. Add the same quantity of pure iron (5.3) as that of the test portion (see 8.2), and the same quantity of the accelerators (5.8) as will be added to the test portion.

8.3.4 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.

8.3.5 Treat the crucible and contents as specified in 8.4.2 and 8.4.3.

8.3.6 Obtain the reading of the blank tests and convert it to milligrams of carbon by means of the calibration graph (see 8.5).

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

8.3.8 The mean blank value ($\bar{m}_1$) is calculated from the two blank values.

8.3.9 The mean blank value and the difference between the two blank values shall both not exceed 0,01 mg of carbon. If these values are abnormally high, investigate and eliminate the source of contamination.

8.4 Determination

8.4.1 Transfer a tin capsule (6.2) into a ceramic crucible (6.3), press the tin capsule lightly against the bottom of the ceramic crucible, add the test portion (see 8.2) and cover with the appropriate mass of the accelerators (5.8) (see 8.3.4).

8.4.2 Place the ceramic crucible and its content on the pedestal post, raise to the combustion position and lock the system. Operate the furnace in accordance with the manufacturer's instructions.

8.4.3 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 graph

8.5.1 Samples having carbon contents between 0,003 % (mass fraction) and 0,01 % (mass fraction)

8.5.1.1 Preparation of the calibration series

Transfer the volumes of sucrose standard solution (5.9) or sodium carbonate standard solution (5.10) given in Table 1 to five 250 ml one-mark volumetric flasks. Dilute to the mark with water (5.1) and mix.

Using the micropipette (6.1), transfer 100 µl of each of the diluted solutions to five tin capsules (6.2) and dry at 90 °C for 2 h.

Allow to cool to room temperature in a desiccator.

Table 1 — Calibration series for carbon mass fractions between 0,003 % and 0,01 %

Volume of the standard solution[a] ml	**Mass of carbon in the diluted solution**, per millilitre mg	**Mass of carbon taken in the tin capsule** mg	**Carbon content in the test portion** % (mass fraction)
0[b]	0	0	0
1,0	0,10	0,010	0,001
2,0	0,20	0,020	0,002
5,0	0,50	0,050	0,005
10,0	1,00	0,100	0,010

[a] Sucrose (5.9) or sodium carbonate (5.10).

[b] Zero member

8.5.1.2 Measurements

Transfer each tin capsule containing sucrose or sodium carbonate into a ceramic crucible (6.3), press the tin capsule lightly against the bottom of the ceramic crucible, add 1,000 g of pure iron (5.3) and cover with the same quantity of the accelerators (5.8) (see 8.3.4) as will be added to the test portion.

Treat the crucible and its content as specified in 8.4.2 and 8.4.3.

8.5.1.3 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.

Prepare a calibration graph by plotting the net reading against the mass of carbon expressed in milligrams corresponding to each member of the calibration series.

8.5.2 Samples having carbon contents between 0,01 % (mass fraction) and 0,1 % (mass fraction)

8.5.2.1 Preparation of the calibration series

Transfer the volumes of sucrose standard solution (5.9) or sodium carbonate standard solution (5.10) indicated in Table 2 to five 50 ml one-mark volumetric flasks. Dilute to the mark with water (5.1) and mix.

Using the micropipette (6.1), transfer 100 µl of each of the diluted solutions to five tin capsules (6.2) and dry at 90 °C for 2 h.

Allow to cool to room temperature in a desiccator.

Table 2 — Calibration series for carbon mass fractions between 0,01 % and 0,1 %

Volume of the standard solution[a] ml	**Mass of carbon in the diluted solution**, per millilitre mg	**Mass of carbon taken in the tin capsule** mg	**Carbon content in the test portion** % (mass fraction)
0[b]	0	0	0
2,0	1,0	0,10	0,010
4,0	2,0	0,20	0,020
10,0	5,0	0,50	0,050
20,0	10,0	1,00	0,100

[a] Sucrose (5.9) or sodium carbonate (5.10).

[b] Zero member

8.5.2.2 Measurements

As specified in 8.5.1.2.

8.5.2.3 Plotting the calibration graph

As specified in 8.5.1.3.

8.5.3 Samples having carbon contents between 0,1 % (mass fraction) and 1,0 % (mass fraction)

8.5.3.1 Preparation of the calibration series

Weigh, to the nearest 0,1 mg, the masses of barium carbonate (5.6) or sodium carbonate (5.7) indicated in Table 3 and transfer to five tin capsules (6.2).

Table 3 — Calibration series for carbon mass fractions between 0,1 % and 1,0 %

Mass of the reagent mg		Mass of carbon taken in the tin capsule mg	Carbon content in the test portion % (mass fraction)
Barium carbonate[a]	**Sodium carbonate**[b]		
0[c]	0[c]	0	0
16,4	8,8	1,0	0,10
32,9	17,7	2,0	0,20
82,1	44,1	5,0	0,50
164,3	88,2	10,0	1,00

[a] Barium carbonate (5.6)

[b] Sodium carbonate (5.7)

[c] Zero member

8.5.3.2 Measurements

Transfer each tin capsule containing barium carbonate or sodium carbonate into a ceramic crucible (6.3), press the tin capsule lightly against the bottom of the ceramic crucible, add 1,000 g of pure iron (5.3) and cover with the same quantity of the accelerators (5.8) (see 8.3.4) as will be added to the test portion.

Treat the crucible and contents as specified in 8.4.2 and 8.4.3.

8.5.3.3 Plotting the calibration graph

As specified in 8.5.1.3.

8.5.4 Samples having carbon contents between 1,0 % (mass fraction) and 4,5 % (mass fraction)

8.5.4.1 Preparation of the calibration series

Weigh, to the nearest 0,l mg, the masses of barium carbonate (5.6) or sodium carbonate (5.7) indicated in Table 4 and transfer to five tin capsules (6.2).

If the weighed barium carbonate cannot be transferred to the tin capsule, it may be placed directly on the bottom of the ceramic crucible.