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

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

Reference number
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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9556 was prepared by Technical Committee ISO/TC 17, *Steel*.

Annexes A, B and C of this International Standard are for information only.

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

1 Scope

This International Standard 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 % (m/m) and 4,5 % (m/m).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 377: 1985, *Wrought steel — Selection and preparation of samples and test pieces.*

ISO 385-1: 1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

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

ISO 1042: 1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 5725: 1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

3 Principle

Combustion of a test portion with 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.

4 Reagents

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

4.1 Water, free from carbon dioxide.

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

4.2 Oxygen, 99,5 % (m/m) minimum.

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

4.3 Pure iron, of known low carbon content less than 0,001 0 % (m/m).

4.4 Suitable solvent, appropriate for washing greasy or dirty test samples, for example, acetone.

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

4.6 Barium carbonate

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

4.7 Sodium carbonate

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

4.8 Accelerator: copper, tungsten-tin mixture or tungsten of known low carbon content less than 0,001 0 % (m/m).

4.9 Sucrose, standard solution, corresponding to 25 g of C per litre.

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

Dissolve in about 100 ml of water (4.1), transfer to a 250 ml one-mark volumetric flask quantitatively, dilute to the mark with water (4.1) and mix.

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

4.10 Sodium carbonate, standard solution, corresponding to 25 g of C per litre.

Weigh, to the nearest 1 mg, 55,152 g of sodium carbonate (4.7), dissolve in about 200 ml of water (4.1), transfer to a 250 ml one-mark volumetric flask quantitatively, dilute to the mark with water (4.1) and mix.

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

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

5 Apparatus

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

All volumetric glassware shall be class A, in accordance with ISO 385-1, 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.

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

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, of known low carbon content less than 0,001 0 % (m/m).

5.3 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.

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

6 Sampling

Carry out sampling in accordance with ISO 377 or appropriate national standards for iron.

7 Procedure

SAFETY INSTRUCTIONS — 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.

7.1 General operating instructions

Purify the oxygen supply using tubes packed with the inert ceramic (attapulgus clay) impregnated with sodium hydroxide (4.11) and magnesium perchlorate (4.5), 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 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, 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 1 mg, approximately 1 g of the test sample for carbon contents less than 1,0 % (m/m) and approximately 0,5 g for carbon contents greater than 1,0 % (m/m).

NOTE — The mass of the test portion may be dependent on the type of instrument used.

7.3 Blank test

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

Transfer the tin capsule (5.2) (see note 1) to the ceramic crucible (5.3), press the tin capsule lightly against the bottom of the crucible. Add the same quantity of pure iron (4.3) as that of the test portion (7.2), and the same quantity of the accelerators (4.8) (see note 2) as will be added to the test portion.

Treat the crucible and contents as specified in the second and third paragraphs of 7.4.

Obtain the reading of the blank tests and convert it to milligrams of carbon by means of the calibration graph (7.5).

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

The mean blank value ($\overline{m}_1$) is calculated from the two blank values (see note 3).

NOTES

1 In cases where the calibration graphs of 7.5.1 or 7.5.2 are applied, use the following prepared capsule.

Prepare the tin capsule; using the micropipette (5.1), transfer 100 µl of water (4.1) to a tin capsule (5.2) and dry at 90 °C for 2 h.

2 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.

3 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.

7.4 Determination

Transfer one tin capsule (5.2) to the ceramic crucible (5.3), press the tin capsule lightly against the bottom of the ceramic crucible, add the test portion (7.2) and cover with the appropriate mass of the accelerators (4.8) (see note 2 of 7.3).

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 Samples having carbon contents between 0,003 % (m/m) and 0,01 % (m/m)

7.5.1.1 Preparation of the calibration series

Transfer the volumes of sucrose standard solution (4.9) or sodium carbonate standard solution (4.10) indicated in table 1 to five 250 ml one-mark volumetric flasks. Dilute to the mark with water (4.1) and mix.

Using the micropipette (5.1), transfer 100 µl of each of the diluted solutions to five tin capsules (5.2) and dry at 90 °C for 2 h.

Cool to room temperature in a desiccator.

Table 1

Volume of the standard solution [(4.9) or (4.10)] 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 % (m/m)
0*)	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

*) Zero member

7.5.1.2 Measurements

Transfer the tin capsule containing sucrose or sodium carbonate to the ceramic crucible (5.3), press the tin capsule lightly against the bottom of the ceramic crucible, add 1,000 g of pure iron (4.3) and cover with the same quantity of the accelerators (4.8) (see note 2 of 7.3) as will be added to the test portion.

Treat the crucible and contents as specified in the second and third paragraphs of 7.4.

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.

Prepare a calibration graph by plotting the net reading against milligrams of carbon for each member of the calibration series.

7.5.2 Samples having carbon contents between 0,01 % (m/m) and 0,1 % (m/m)

7.5.2.1 Preparation of the calibration series

Transfer the volumes of sucrose standard solution (4.9) or sodium carbonate standard solution (4.10) indicated in table 2 to five 50 ml one-mark volumetric flasks. Dilute to the mark with water (4.1) and mix.

Using the micropipette (5.1), transfer 100 µl of each of the diluted solutions to five tin capsules (5.2) and dry at 90 °C for 2 h.

Cool to room temperature in a desiccator.

Table 2

Volume of the standard solution [(4.9) or (4.10)] 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 % (*m*/*m*)
0*)	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

*) Zero member

7.5.2.2 Measurements

As specified in 7.5.1.2.

7.5.2.3 Plotting the calibration graph

As specified in 7.5.1.3.

7.5.3 Samples having carbon contents between 0,1 % (*m*/*m*) and 1,0 % (*m*/*m*)

7.5.3.1 Preparation of the calibration series

Weigh, to the nearest 0,1 mg, the masses of barium carbonate (4.6) or sodium carbonate (4.7) indicated in table 3 and transfer to five tin capsules (5.2).

Table 3

Mass of the reference material mg		Mass of carbon taken in the tin capsule mg	Carbon content in the test portion % (*m*/*m*)
Barium carbonate (4.6)	Sodium carbonate (4.7)		
0*)	0*)	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

*) Zero member

7.5.3.2 Measurements

Transfer the tin capsule containing barium carbonate or sodium carbonate to the ceramic crucible (5.3), press the tin capsule lightly against the bottom of the ceramic crucible, add 1,000 g of pure iron (4.3) and cover with the same quantity of the accelerators (4.8) (see note 2 of 7.3) as will be added to the test portion.

Treat the crucible and contents as specified in the second and third paragraphs of 7.4.

7.5.3.3 Plotting the calibration graph

As specified in 7.5.1.3.

7.5.4 Samples having carbon contents between 1,0 % (*m*/*m*) and 4,5 % (*m*/*m*)

7.5.4.1 Preparation of the calibration series

Weigh, to the nearest 0,1 mg, the masses of barium carbonate (4.6) or sodium carbonate (4.7) indicated in table 4 and transfer to five tin capsules (5.2).

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

Table 4

Mass of the reference material mg		Mass of carbon taken in the tin capsule mg	Carbon content in the test portion % (*m*/*m*)
Barium carbonate (4.6)	Sodium carbonate (4.7)		
0*)	0*)	0	0
82,1	44,1	5,0	1,0
164,3	88,2	10,0	2,0
246,4	132,3	15,0	3,0
369,7	198,6	22,5	4,5

*) Zero member

7.5.4.2 Measurements

Transfer the tin capsule containing barium carbonate or sodium carbonate to the ceramic crucible (5.3), press the tin capsule lightly against the bottom of the ceramic crucible, add 0,500 g of pure iron (4.3) and cover with the same quantity of the accelerators (4.8) (see note 2 of 7.3) as will be added to the test portion.

Treat the crucible and contents as specified in the second and third paragraphs of 7.4.

7.5.4.3 Plotting the calibration graph

As specified in 7.5.1.3.

8 Expression of results

8.1 Method of calculation

Convert the analyser reading of the test portion to milligrams of carbon (m_0) by means of the calibration graph (7.5).

The carbon content, expressed as a percentage by mass, w_c (%), is given by the equation:

$$\frac{(m_0 - \bar{m}_1)}{m \times 10^3} \times 100$$

$$= \frac{(m_0 - \bar{m}_1)}{10m}$$

where

m_0 is the mass, expressed in milligrams of carbon, in the test portion;

$\bar{m}_1$ is the mass, expressed in milligrams of carbon, in the blank test (7.3);

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

8.2 Precision

A planned trial of this method was carried out by 22 laboratories, at 12 levels of carbon, each laboratory making three determinations of carbon content at each level (see notes 1 and 2).

The test samples used and mean results obtained are listed in table A.1.

The results obtained were treated statistically in accordance with ISO 5725.

The data obtained showed a logarithmic relationship between carbon content and repeatability (r) and reproducibility (R and R_w) of the test results (see note 3) as summarized in table 5. The graphical representation of the figures is given in annex B.

Table 5

Carbon content	Repeatability	Reproducibility	
% (m/m)	r	R	R_w
0,003	0,000 53	0,001 19	0,000 77
0,005	0,000 69	0,001 60	0,001 02
0,01	0,000 99	0,002 40	0,001 50
0,02	0,001 42	0,003 59	0,002 20
0,05	0,002 29	0,006 12	0,003 65
0,1	0,003 29	0,009 17	0,005 36
0,2	0,004 72	0,013 7	0,007 85
0,5	0,007 62	0,023 4	0,013 0
1,0	0,011 0	0,035 1	0,019 1
2,0	0,015 7	0,052 6	0,028 0
4,5	0,024 0	0,084 4	0,043 8

NOTES

1 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725, i.e. one operator, same apparatus, identical operating conditions, same calibration, and a minimum period of time.

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.

3 From the two values obtained on day 1 the repeatability (r) and reproducibility (R) were calculated using the procedure specified in ISO 5725. From the first value obtained on day 1 and the value obtained on day 2, the within-laboratory reproducibility (R_w) was calculated.

9 Test report

The test report shall include the following information:

a) all information necessary for the identification of the sample, the laboratory and the date of the analysis;

b) the method used, by reference to this International Standard;

c) the results, and the form in which they are expressed;

d) any unusual features noted during the determination;

e) any operation not specified in this International Standard, or any optional operation which may have influenced the results.

Annex A
(informative)

Additional information on the international co-operative tests

Table 5 was derived from the results of international analytical trials carried out in 1985 on seven steel samples and five iron samples in eight countries involving 22 laboratories.

The results of the trials were reported in document 17/1 N 668, April 1986. The graphical representation of the precision data is given in annex B.

The test samples used are listed in table A.1.

Table A.1

Sample		Carbon content % (*m/m*)		
		Certified	Found	
			$\bar{w}_1$	$\bar{w}_2$
EURO B 097-1	High-purity iron	⩽ 0,002	0,001 4	0,001 3
JSS 001-2	High-purity iron	0,004 7	0,004 5	0,004 4
NBS SRM 365	Electrolytic iron	0,006 8	0,007 5	0,007 5
BCS 431/1	Carbon steel	0,026	0,025 8	0,025 7
JSS 171-3	Mild steel	0,042	0,041 2	0,040 9
NBS SRM 15g	Carbon steel	0,094	0,092 8	0,092 6
JSS 030-4	Carbon steel	0,18	0,183	0,182
EURO F 080-1	Carbon steel	0,452	0,457	0,456
NBS SRM 14f	Carbon steel	0,753	0,760	0,760
EURO B 063-1	Carbon steel	1,26	1,267	1,266
NBS SRM 3d	White iron	2,54	2,556	2,559
JSS 110-7	Pig iron	4,12	4,095	4,095

$\bar{w}_1$: General mean of within a day

$\bar{w}_2$: General mean of between days

Annex B
(informative)

Graphical representation of precision data

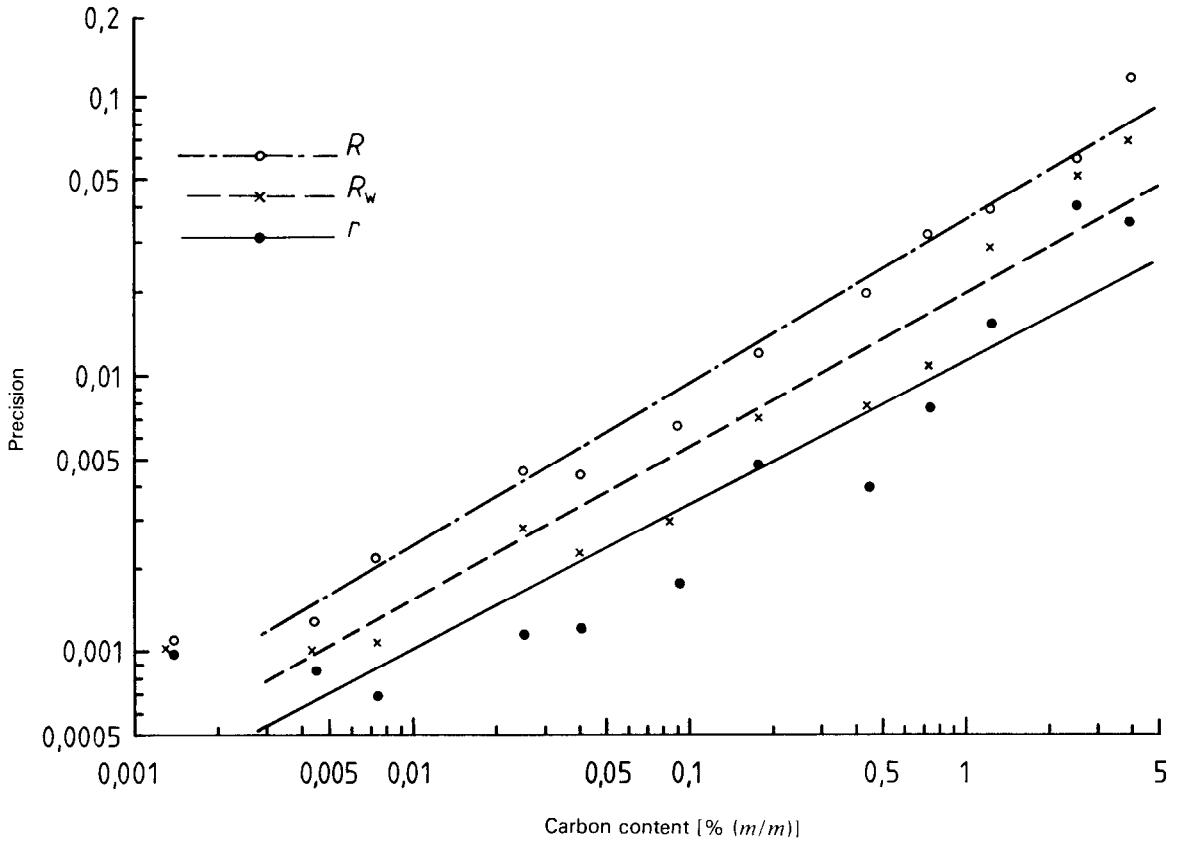

Figure B.1 — Logarithmic relationship between carbon content [% (m/m)] and repeatability (r) or reproducibility (R and R_w)

Annex C
(informative)

Features of commercial high-frequency induction furnaces and infrared carbon analysers

C.1 Source of oxygen, 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 28 kN/m^2.

C.2 Purifying unit, containing the inert ceramic impregnated with sodium hydroxide in a carbon dioxide absorbing tube, and the magnesium perchlorate in a dehydration tube.

C.3 Flowmeter, capable of measuring a flow of oxygen of 0 to 4 l/min.

C.4 High-frequency induction furnace

C.4.1 The combustion furnace consists of an induction coil and a high-frequency generator. The furnace chamber consists of a silica tube (e.g. 30 mm to 40 mm in outer diameter, 26 mm to 36 mm in inner diameter, 200 mm to 220 mm in length) which fits inside the induction coil. This tube has metal plates at the top and bottom which are sealed to the tube by O-rings. Gas inlet and outlet points are made through the metal plates.

C.4.2 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 have been used. The power from the generator is fed to the induction coil which surrounds the silica furnace tube and is usually air-cooled.

C.4.3 The crucible containing the sample, flux 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.

C.4.4 The induction coil diameter, the number of turns, the furnace chamber geometry and the power of the generator determine the degree of coupling which can occur. These factors are determined by the instrument manufacturer.

C.4.5 The temperature attained during the combustion depends in part on the factors indicated in C.4.4, but also on the characteristics of the metal in the crucible, the form of the test portion and the mass of materials. Certain of these factors may be varied to some extent by the operator.

C.5 Dust collector, capable of collecting metal oxide dust in a current of oxygen from the furnace.

C.6 Desulfurization tube, consisting of a heated oxidation tube containing a platinum foil or platinized silica and a sulfur trioxide collector containing cellulose cotton.

C.7 Infrared gas analyser

C.7.1 In most instruments the gaseous products of combustion are transferred to the analyser system in a continuous flow of oxygen. 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 percentage concentration of carbon.

C.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.

C.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 the standard or test portion for automatic correction of the read-out. 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.

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Descriptors : steels, iron, chemical analysis, determination of content, carbon, infrared spectra, absorption spectra.

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