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

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

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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 main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

- type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;
- type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;
- type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 10719, which is a Technical Report of type 2, was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

During the 12th ISO/TC 17/SC 1 meeting held in Sydney in 1988, WG 20 was created to study the determination of non-combined carbon content in irons by the infrared absorption method after combustion in an induction furnace.

Two methods were proposed: one was provided by the USA and the other was taken from EURONORM 38-83.

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Finally, after preliminary tests, a document was prepared on the basis of the method from the USA as a first working draft. A test programme for the international cooperative test was carried out in accordance with this document.

The results showed that the most important point was sampling. The worst results were obtained with fine dust samples and the best with big chips (see annex C).

At the 13th ISO/TC 17/SC 1 meeting held in Madrid in 1990, it was decided that WG 20 should continue with precision testing using the same method (2nd draft) and, taking note of the remarks about sampling, with a new choice of samples specifying the size of the chips and precautions for transport conditions.

A second test programme for an international cooperative test was then carried out.

In spite of all the precautions taken for the choice of certified reference material (CRM), the size of the chips and the transport conditions, it appeared impossible to obtain results better than those of the first trial.

Probably, the best results would be obtained on solid samples, but there are no CRMs in this form. This opinion had already been expressed by some participating laboratories in the first trial (see annex D).

At the 14th ISO/TC 17/SC 1 meeting held in London in 1992, after discussion, it was decided that the method and work of WG 20 and associated studies would be published as a Technical Report.

Annexes A, B, C, D and E of this Technical Report are for information only.

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

1 Scope

This Technical Report describes an infrared absorption method after combustion in an induction furnace for the determination of the non-combined carbon content in steel and iron.

The method is applicable to non-combined carbon contents between 0,3 % (m/m) and 3,0 % (m/m).

Elements ordinarily present do not interfere. However, some alloyed cast irons, when extensively heat treated, yield carbides that are not soluble when using this method and may give high values for non-combined carbon.

2 Normative references

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

ISO 377-2:1989, *Selection and preparation of samples and test pieces of wrought steels — Part 2: Samples for the determination of the chemical composition.*

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

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

ISO 9556:1989, *Steel and iron — Determination of total carbon content — Infrared absorption method after combustion in an induction furnace.*

3 Definition

For the purposes of this Technical Report, the following definition applies.

3.1 non-combined carbon: Carbon which is not dissolved by the procedure specified in 8.4.1 and is also known as graphitic carbon.

4 Principle

Decomposition of a test portion with nitric acid in the presence of methanol and treatment with hydrofluoric acid.

Removal of the non-combined carbon by filtering through a glass-fibre filter.

Combustion of the glass-fibre filter containing the non-combined carbon with the pure iron and accelerator, 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 water with a low content of organic matter, i.e. grade 2 or grade 1 water as specified in ISO 3696.

5.1 Water, free from carbon dioxide.

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

5.2 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 E), 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 % (*m/m*).

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

5.5 Methanol, 99,5 % (*V/V*) minimum.

5.6 Barium carbonate.

Dry barium carbonate [minimum assay: 99,5 % (*m/m*)] 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 assay: 99,5 % (*m/m*)] 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 % (*m/m*).

5.9 Nitric acid, ρ about 1,40 g/ml.

5.10 Hydrofluoric acid, ρ about 1,15 g/ml.

5.11 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1 + 1.

5.12 Sodium hydroxide, 120 g/l solution.

Cautiously dissolve 60 g of sodium hydroxide in about 200 ml of water (5.1). When dissolution is complete, cool, dilute to 500 ml and store in a plastics bottle.

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

5.14 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 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 E.

6.1 Filter, made of glass fibre, 47 mm in diameter, pore size 0,3 µm.

6.2 Vacuum filtering apparatus, for use with 47 mm glass-fibre filters and suitable for use with acids.

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.

7 Sampling

Carry out sampling in accordance with ISO 377-2 and appropriate national standards for iron (see note 1).

NOTE 1 For cast iron and steel, use drillings with lengths of more than 2 mm. For ductile iron, use solid portions, approximately 10 mm × 10 mm × 0,3 mm (see ASTM E 351:1989, *Standard Test Methods for Chemical Analysis of Cast Iron — All types*).

8 Procedure

SAFETY INSTRUCTIONS

a) 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 must be removed effectively from the apparatus, since a high concentration of oxygen in a confined space can present a fire hazard.

b) Take care not to mix methanol with concentrated nitric acid because of the risks of explosion.

8.1 General operating instructions

Purify the oxygen supply (5.2) using tubes packed with the inert ceramic (5.14) impregnated with sodium hydroxide (5.12) and magnesium perchlorate (5.13), and maintain a quiescent flow rate whilst on standby. Maintain a glass-fibre filter or a stainless steel filter-screen 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.

When the main supply is switched on after being out of action for any length of time, allow the equipment to stabilize for the time recommended by the equipment manufacturers.

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 of iron 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 of ISO 9556:1989.

Adjust the reading of the instrument to the certified value (see note 2).

NOTE 2 This adjustement should be made before the calibration specified in 8.5. It cannot replace or correct the calibration.

8.2 Test portion

Degrease the test sample, if necessary, by washing in a suitable solvent (5.4). Cautiously evaporate the last traces of the solvent by warming.

Weigh, to the nearest 1 mg, approximately 0,50 g of the test sample for non-combined carbon contents greater than 1,0 % (*m/m*) and approximately 1,0 g for non-combined carbon contents not more than 1,0 % (*m/m*).

8.3 Blank test

In parallel with the determination and following the same procedure, carry out two blank tests using the same quantities of all the reagents, including the same quantity of the pure iron (5.3) as used for the test portion (8.2).

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

The mean blank value (m_1) is calculated from the two blank values (see note 3).

NOTE 3 The mean blank value and the difference between the two blank values shall both not exceed 0,15 mg of carbon. If these values are abnormally high, investigate and eliminate the source of contamination.

8.4 Determination

8.4.1 Dissolution of the test portion

Place the test portion (8.2) in a 250 ml beaker. Add 25 ml of methanol (5.5), then 50 ml of water (5.1) and lastly 5 ml of nitric acid (5.9). Immediately cover the beaker with a watch glass and allow to stand for 12 h to 16 h at room temperature.

Add 20 ml of nitric acid (5.9) and allow to stand until further action ceases. Place the beaker on a hotplate heated to 50 °C to 60 °C. When vigorous action ceases, add 4 to 5 drops of hydrofluoric acid (5.10). Continue heating until dissolution is completed. If necessary, add water (5.1) to maintain the original volume.

8.4.2 Filtration of the non-combined carbon

Place two glass-fibre filters (6.1) in a vacuum filtering apparatus (6.2) and moisten with water (5.1). Filter the solution obtained in 8.4.1 with very gentle suction. Transfer the non-combined carbon to the filter and police the beaker thoroughly. Wash the sides of the funnel to deposit all non-combined carbon on the glass-fibre filter disc.

Wash the filter disc once with hydrochloric acid (5.11), twice with hot water (5.1), three times with hot sodium hydroxide solution (5.12), twice with hot hydrochloric acid and five times with hot water, in the order given. Finally, wash with methanol (5.5) to remove any non-combined carbon from the walls of the funnel.

Turn off the suction and remove the top part of the filter assembly. Remove the top filter disc from the filter support by using a small pair of tweezers, fold it in octavo and place in a ceramic crucible (6.3). Remove the bottom filter disc from the filter support and use it to wipe off any non-combined carbon from the filter apparatus. Fold the filter disc in octavo and place it in the same crucible.

Dry the crucible containing the filters for 2 h at 105 °C, and store in a desiccator.

8.4.3 Measurements

Add 1,000 g of the pure iron (5.3) and the appropriate mass of accelerator (see note 4) to the ceramic crucible containing the dried filters described in 8.4.2.

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.

NOTE 4 The quantity of accelerator (5.8) will depend on the individual characteristics of the instrument. The amount used should be sufficient for complete combustion.

8.5 Establishing the calibration graph

8.5.1 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 1 and transfer to four ceramic crucibles (6.3).

Add two glass-fibre filters (6.1) to the ceramic crucible (6.3) containing barium carbonate (5.6) or sodium carbonate (5.7). Then add 1,000 g of the pure iron (5.3) and cover with the same quantity of the accelerator (5.8) as will be used for the test portion.

Table 1

Mass of the reference material mg		**Mass of carbon taken in the ceramic crucible** mg	**Corresponding non-combined carbon content in a 0,5 g test portion** % *(m/m)*
Barium carbonate (5.6)	Sodium carbonate (5.7)		
0[1]	0[1]	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

1) Zero member.

8.5.2 Measurements

Treat the crucible and contents (see 8.5.1) as specified in 8.4.3.

8.5.3 Plotting the calibration graph

Obtain the net reading by subtracting the reading of the zero member (see table 1) from that of each member of the calibration series.

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

9 Expression of results

9.1 Method of calculation

Convert the analyser reading of the test portion to milligrams of carbon (m_0) by means of the calibration graph (see 8.5).

The non-combined carbon content, w_C, expressed as a percentage by mass, is given by the equation

$$w_C = \frac{(m_0 - m_1)}{m \times 10^3} \times 100$$

$$= \frac{(m_0 - m_1)}{10m}$$

where

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

m_1 is the mass, expressed in milligrams, of carbon in the blank test (see 8.3);

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

9.2 Precision

A planned trial of this method was carried out by 12 laboratories, using 10 levels of non-combined carbon contents, each laboratory making three determinations of non-combined carbon on each level (see notes 5 and 6).

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 non-combined carbon content and repeatability (r) and reproducibility (R and R_w) of the test results (see note 7) as summarized in table 2. The graphical representation of the data is shown in figure B.1.

Table 2

Non-combined carbon content	Repeatability	Reproducibility	
% (m/m)	r	R	R_w
0,30	0,049 1	0,118	0,055 8
0,50	0,055 9	0,148	0,065 8
1,00	0,066 7	0,200	0,082 5
2,00	0,079 6	0,271	0,103
3,00	0,088 3	0,324	0,118

NOTES

5 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.

6 The third determination was carried out at a different time (on a different day) by the same operator as in note 5 using the same apparatus with a new calibration.

7 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.

10 Notes on procedure

10.1 Preliminary blank tests

To test water (5.1) and the different reagents, carry out two preliminary blank tests at the same time, following the same procedure and using the same quantities of all the reagents, including the same quantity of the pure iron (5.3) as used for the test portion (8.2).

All the preliminary blank values shall be equal to or less than 0,15 mg of carbon. If they are not, it is necessary to take and select other reagents and water and to repeat the preliminary blank test.

10.2 Preliminary tests on certified reference materials

After selecting reagents and water, and before carrying out the tests, make preliminary tests using some established certified reference materials.

Carry out two determinations under repeatability conditions, following the same procedure and using the same quantities of all the reagents as used for the test portion.

The difference between the mean value and the certified value of the certified reference material used should be equal to or less than $2{,}0 \times r/2{,}8$, and the difference between the two determinations should be equal to or less than r (the value of r is a function of the non-combined carbon content, see table 2). If they are not, the preliminary test should be repeated until these criteria are met.

11 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 Technical Report;

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 Technical Report, or any optional operation which may have influenced the results.

Annex A
(informative)

Additional information on the international co-operative tests

Table 2 was derived from the results of the second international analytical trials carried out in 1991 on one steel sample and nine iron samples in seven countries involving 12 laboratories.

The results of the trials were reported in document ISO/TC 17/SC 1 N 925, February 1992. The graphical representation of the precision data is given in annex B.

The smoothed curve of reproducibility is beyond the acceptable line which is described in documents ISO/TC 17/SC 1 N 967 and N 976.

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

Table A.1

Sample	Total carbon content % (m/m)	Non-combined carbon content % (m/m)			Precision data 1)		
		Certified	Found		Repeatability	Reproducibility	
			$\bar{w}_{C,1}$	$\bar{w}_{C,2}$	r	R	R_w
NIST 342a (Nodular cast iron)	1,9	1,38	1,419	1,412	0,080 9	0,249	0,118
BCS 172/3 [Cast iron (Ni–Cr–Cu–Mo)]	2,2	1,55	1,546	1,551	0,071 0	0,278	0,066 1
ECRM 483-1 (Cast iron)	2,5	1,65	1,685	1,700	0,095 7	0,298	0,116
CTIF FG20 (Cast iron)	2,5	1,924 2)	1,953	1,955	0,078 9	0,276	0,074 0
NIST 107c [Cast iron (Ni–Cr–Mo)]	3,0	1,98	2,132	2,133	0,087 2	0,405	0,090 7
BCS 206/2 [Cast iron (high Si, P)]	2,5	2,4 3)	2,406	2,408	0,017 9	0,166	0,049 6
ECRM 428-1 (Cast iron)	2,8	2,33	2,313	2,319	0,108	0,224	0,116
NIST 4k (Cast iron)	3,2	2,65	2,642	2,652	0,104	0,286	0,148
NIST 122h (Cast iron)	3,5	2,82	2,882	2,884	0,179	0,397	0,254
Steel	1,4	0,395 2)	0,383	0,388	0,052 6	0,120	0,068 5

$\bar{w}_{C,1}$: general mean within a day

$\bar{w}_{C,2}$: general mean between days

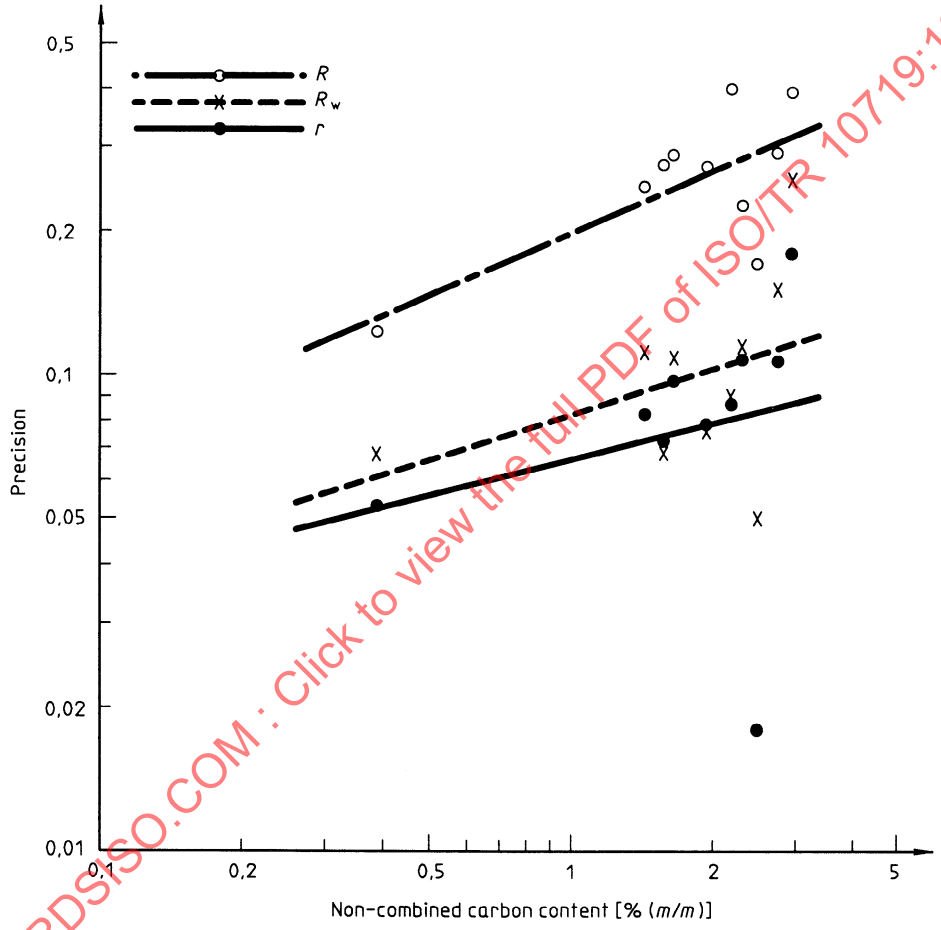
1) For non-combined carbon content.

2) Mean value of a preliminary round robin test (non-certified value).

3) Non-certified value.

Annex B
(informative)

Graphical representation of precision data



$$\lg r = 0{,}255\,3 \ \lg \overline{w}_{C,1} - 1{,}175\,7$$

$$\lg R_w = 0{,}325\,8 \ \lg \overline{w}_{C,2} - 1{,}083\,4$$

$$\lg R = 0{,}439\,6 \ \lg \overline{w}_{C,1} - 0{,}698\,6$$

where

$\overline{w}_{C,1}$ is the average non-combined carbon content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{C,2}$ is the average non-combined carbon content, expressed as a percentage by mass, obtained between days.

Figure B.1 — Logarithmic relationship between non-combined carbon content (w_C) and repeatability (r) or reproducibility (R and R_w)

Annex C
(informative)

Comparison between the results of 1st and 2nd trials

Test sample number	Name of the sample	Non-combined carbon content % (*m/m*)			Precision data[1]	
		Certified	Found $(\overline{w}_{C,1} + \overline{w}_{C,2})/2$		Reproducibility R	
			1st trial	2nd trial	1st trial	2nd trial
20-1	ECRM 430-1	2,338	2,336		0,240	
20-2[2]	Pig iron	0,289	0,519		1,408	
20-3,20	Steel	0,395[3]	0,393	0,385	0,191	0,120
20-4	P-405	2,813[3]	2,794		0,330	
20-5,17	ECRM 428-1	2,33	2,384	2,316	0,158	0,224
20-6	NIST 5l	1,98	1,991		0,233	
20-7	NIST 8b	2,37	2,361		0,234	
20-8,13	ECRM 483-1	1,65	1,688	1,692	0,298	0,298
20-9	CTIF FG10	1,647[3]	1,694		0,329	
20-10,14	CTIF FG20	1,924[3]	1,907	1,954	0,122	0,276
20-11	NIST 342a	1,38		1,416		0,249
20-12	BCS 172-3	1,55		1,549		0,278
20-15	NIST 107c	1,98		2,133		0,405
20-16	BCS 206-2	2,4[4]		2,407		0,166
20-18	NIST 4k	2,65		2,647		0,286
20-19	NIST 122h	2,82		2,833		0,397

1) For non-combined carbon content.

2) Fine dust sample.

3) Mean value of a preliminary round robin test (non-certified value).

4) Non-certified value.

Annex D
(informative)

Effect of fine material in a sample on the determination of non-combined carbon content

During the international cooperative tests carried out by ISO/TC 17/SC 1/WG 20, the following information was submitted from the USA (see document ISO/TC 17/SC 1 N 925 "Report of WG 20" for details).

D.1 Comments and test results

It is possible that the variability in results obtained during the WG 20 tests might be caused by the presence of fine loose non-combined carbon. The following determinations, which were conducted on 13 samples of NIST 122i (cast iron in chip form) supplied for homogeneity testing for total carbon, tend to support that view.

The samples were taken from large cans of NIST 122i and were identified by a can number, and "T" for samples taken from the top of a can or "B" for samples from the bottom of a can.

D.1.1 Determination of total carbon in separate samples

Each of the 13 samples was run 12 times on a Leco CS-44 instrument using a 0,4 g test portion and 2,2 g of tungsten accelerator. The results are given in table D.1.

There is a wide range of values for the carbon content, although the mean for the six "T" samples is the same as the mean for the seven "B" samples: 3,494 % (*m/m*) in each case, which is identical to the mean for all 156 determinations.

D.1.2 Determination of total carbon in a composite sample after sieving

A composite sample was prepared by combining 10 g from each of the 13 samples. The 130 g sample was then sieved through a screen (40 mesh) while shaking and tapping for 10 min. Approximately 0,24 g of material passed through the screen.

D.2 Analysis of the composite sample from which the fine material was removed

Total carbon content (13 determinations)

3,481 % (*m/m*)

3,454 % (*m/m*)

3,452 % (*m/m*)

3,457 % (*m/m*)

3,432 % (*m/m*)

3,454 % (*m/m*)

3,431 % (*m/m*)

3,419 % (*m/m*)

3,405 % (*m/m*)

3,408 % (*m/m*)

3,361 % (*m/m*)

3,456 % (*m/m*)

3,415 % (*m/m*)

Mean = 3,433 % (*m/m*)

Standard deviation = 0,031 3 % (*m/m*)

Coefficient of variation = 0,912 %

D.3 Analysis of fine material

Total carbon content = 31,8 % (*m/m*)

D.4 Calculated total carbon content of the composite sample before sieving

Mass of carbon in the fine material removed by sieving = 0,24 × 0,318 = 0,076 g

Contribution to the carbon content of the total 130 g sample $= \frac{0{,}076 \times 100}{130} = 0{,}058$ % (*m/m*)

Total carbon content in the composite sample before sieving = 3,433 + 0,058 = 3,491 % (*m/m*)

This correlates very well with the value of 3,494 % (*m/m*) obtained for the mean of 156 determinations of the 13 separate samples.

D.5 Conclusions

This evidence strongly suggests that the presence of excessive non-combined carbon [31,8 % (*m/m*) in this case] in the fine material affects the total carbon homogeneity and would similarly affect the non-combined carbon homogeneity, resulting in the wide spread of results obtained during the WG 20 trials.

Table D.1 — Determination of carbon content in composite sample NIST 122i [% (*m/m*)]

Run	Sample													
	1T	1B	2T	2B	3T	3B	4T	4B	5T	5B	6T	6B	7B
1	3,506	3,406	3,571	3,598	3,533	3,560	3,457	3,542	3,500	3,510	3,504	3,594	3,533
2	3,518	3,448	3,514	3,571	3,572	3,516	3,493	3,510	3,506	3,500	3,515	3,549	3,525
3	3,470	3,472	3,523	3,527	3,589	3,539	3,550	3,539	3,461	3,463	3,521	3,516	3,548
4	3,451	3,409	3,564	3,528	3,579	3,544	3,470	3,486	3,450	3,447	3,526	3,569	3,521
5	3,433	3,403	3,451	3,509	3,556	3,557	3,409	3,524	3,495	3,454	3,451	3,521	3,470
6	3,470	3,461	3,560	3,543	3,540	3,501	3,475	3,519	3,473	3,454	3,510	3,584	3,509
7	3,465	3,452	3,553	3,544	3,593	3,488	3,473	3,542	3,460	3,492	3,493	3,499	3,530
8	3,490	3,425	3,543	3,521	3,490	3,489	3,485	3,502	3,501	3,526	3,542	3,589	3,480
9	3,448	3,440	3,560	3,485	3,554	3,537	3,533	3,455	3,477	3,433	3,419	3,506	3,367
10	3,500	3,416	3,525	3,467	3,471	3,457	3,501	3,533	3,488	3,336	3,424	3,440	3,449
11	3,375	3,442	3,450	3,525	3,471	3,051	3,457	3,471	3,450	3,475	3,432	3,491	3,423
12	3,398	3,384	3,545	3,554	3,530	3,484	3,435	3,498	3,479	3,444	3,366	3,450	3,432
Average	3,460	3,430	3,530	3,531	3,540	3,515	3,478	3,510	3,478	3,461	3,475	3,526	3,482
SD[1]	0,042 8	0,026 7	0,041 0	0,035 4	0,043 0	0,032 5	0,038 8	0,028 6	0,019 9	0,048 8	0,054 9	0,052 2	0,055 4
CV (%) [2]	1,239 2	0,779 3	1,162 7	1,002 6	1,215 9	0,925 4	1,116 9	0,812 5	0,574 5	1,412 7	1,581 7	1,480 8	0,591 8

NOTE — Samples from some of the 18 large cans of NIST 122i chips. The "T" samples are from the top of the cans and the "B" samples are from the bottom of the cans.

[1] SD: standard deviation

[2] CV: coefficient of variation

Annex E
(informative)

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

E.1 Source of oxygen, fitted with a fine regulating value 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.

E.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.

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

E.4 High-frequency induction furnace.

E.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.

E.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.

E.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.

E.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.

E.4.5 The temperature attained during the combustion depends in part on the factors indicated in E.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.

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

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

E.7 Infrared carbon analyser.

E.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.

E.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.

E.7.3 Electronic controls are usually provided for adjusting the instrument zero, compensating for the blank, adjusting the slope of the calibration line and