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

Aciers et fontes — Dosage du carbone et du soufre totaux — Méthode par absorption dans l'infrarouge après combustion dans un four à induction (méthode pratique)

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Foreword

SO (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 15350 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

Annexes A to D of this International Standard are for information only.

INTERNATIONAL STANDARD **ISO 15350:2000(E)**

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

1 Scope

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

The method is applicable to carbon contents of mass fraction between 0,005 % and 4,3 % and to sulfur contents of mass fraction between 0,000 5 % and 0,33 %.

This method is intended to be used in normal production operations and is intended to meet all generally accepted, good laboratory practices of the type expected by recognized laboratory accreditation agencies. It uses commercially available equipment, is calibrated and calibration verified using steel and iron certified reference materials, and its performance is controlled using normal statistical process control (SPC) practices.

This method can be used in the single element mode, i.e., determination of carbon and sulfur independently or in the simultaneous mode, i.e., determination of carbon and sulfur concurrently.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard 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 437:1982, *Steel and cast iron — Determination of total carbon content — Combustion gravimetric method*.

ISO 4934:1980, *Steel and cast iron — Determination of sulfur content — Gravimetric method*.

ISO 4935:1989, *Steel and iron — Determination of sulfur content — Infrared absorption method after combustion in an induction furnace*.

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

ISO 10701:1994, *Steel and iron — Determination of sulfur content — Methylene blue spectrophotometric method*.

ISO 13902:1997, *Steel and iron — Determination of high sulfur content — Infrared absorption method after combustion in an induction furnace*.

ISO 14284:1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*.

3 Principle

3.1 Carbon

The carbon is converted to carbon monoxide and/or carbon dioxide by combustion in a stream of oxygen. Measurement is by infrared absorption of the carbon monoxide and carbon dioxide carried by a current of oxygen.

3.2 Sulfur

The sulfur is converted to sulfur dioxide by combustion in a stream of oxygen. Measurement is by infrared absorption of the sulfur dioxide carried by a current of oxygen.

4 Reagents

4.1 Acetone, the residue after evaporation shall have a mass fraction less than 0,000 5 %.

4.2 Cyclohexane, the residue after evaporation shall have a mass fraction less than 0,000 5 %.

4.3 Inert ceramic, attapulques clay impregnated with sodium hydroxide and having particle sizes from 0,7 mm to 1,2 mm for absorption of carbon dioxide.

4.4 Pure iron, used as an accelerator, 0,4 mm to 0,8 mm size with carbon and sulfur contents with a mass fraction of less than 0,001 % respectively.

4.5 Magnesium perchlorate, reagent grade, having particle size from 0,7 mm to 1,2 mm for absorption of moisture.

4.6 Oxygen, ultra high purity (mass fraction minimum 99,5 %)

An oxidation catalyst [copper(II) oxide or platinum] tube heated to 600 °C followed by suitable carbon dioxide and water absorbents shall be used when the presence of organic contaminants is suspected in the oxygen.

4.7 Platinum or platinized silica, heated to 350 °C for the conversion of carbon monoxide to carbon dioxide.

4.8 Accelerator, copper, tungsten-tin or tungsten for carbon determination and tungsten for sulfur determination, 0,4 mm to 0,8 mm size with carbon and sulfur contents of mass fraction less than 0,001 % and 0,000 5 % respectively.

4.9 Cellulose cotton, for the collection of sulfur trioxide

4.10 Steel and iron certified reference materials (CRMs), all reference materials used for calibration and calibration verification shall be certified by internationally-recognized bodies and validated by adequate performance on one or more national or international interlaboratory test programmes. Preference shall be given to materials that were certified using referee methods, e.g. ISO 437 and ISO 9556 for carbon, and ISO 4934, ISO 4935, ISO 10701 and ISO 13902 for sulfur, traceable to SI units as opposed to those based on other certified reference materials.

4.11 Steel and iron reference materials (RMs), those used for statistical process control of the method need not be certified, but adequate homogeneity data shall be available, either from the certifying body or from the laboratory that uses the material, in order to ensure the validity of the control data generated.

5 Apparatus

Ordinary laboratory equipment plus the following shall be used.

5.1 C and/or S determinator, consisting of an IR energy source, a separate measuring chamber and reference chamber, and a diaphragm acting as one plate of a parallel plate capacitor.

5.2 Ceramic crucible, as specified by the manufacturer of the instrumentation used and capable of withstanding combustion in an induction furnace without evolving carbon- and sulfur-containing chemicals so that achieving and maintaining blank values within specification is possible.

NOTE Carbon and sulfur contamination can usually be removed by igniting the crucibles in an electric furnace in air for not less than 40 min at 1 000 °C or not less than 15 min at 1 350 °C. After treatment, remove them from the heat, allow them to cool for 2 min to 3 min on an appropriate clean heat-resistant tray and then store them in a desiccator.

5.3 Crucible tongs, capable of handling recommended crucibles (5.2).

6 Test method

This test method is written for use with commercial analysers, equipped to carry out the included operations automatically and calibrated using steels and irons of known carbon and sulfur contents.

The analyser used will be satisfactory if it meets the criteria listed in clause 8.

6.1 Infrared (IR) absorption for carbon — Method A

The amount of carbon dioxide is measured by infrared absorption. Carbon dioxide (CO_2) absorbs IR energy at a precise wavelength within the IR spectrum. Energy of this wavelength is absorbed as the gas passes through a cell body in which the IR energy is transmitted. All other IR energy is prevented from reaching the detector by use of a precise wavelength filter. Thus, the absorption of IR energy can only be attributed to CO_2 and its concentration is measured as changes in energy at the detector. One cell is used as both a reference and a measuring chamber. Total carbon, as CO_2, is monitored and measured over a period of time. See Figure A.1.

6.2 Infrared (IR) absorption for carbon — Method B

During specimen combustion, the flow of CO_2 with its oxygen gas carrier is routed through the measuring chamber (see 5.1) while oxygen alone passes through the reference chamber. Energy from the IR source passes through both chambers, simultaneously arriving at the diaphragm (capacitor plate). Part of the IR energy is absorbed by the CO_2 present in the measuring chamber while none is absorbed passing through the reference chamber. This creates an IR energy imbalance reaching the diaphragm, thus distorting it. This distortion alters the fixed capacitance creating an electric signal change that is amplified for measurement as CO_2. Total carbon, as CO_2, is monitored and measured over a period of time. See Figure A.2.

6.3 Infrared (IR) absorption for carbon — Method C, closed loop

The combustion is performed in a closed loop, where CO and CO_2 are detected in the same infrared cell. Each gas is measured using a solid state energy detector. Filters are used to pass the appropriate IR wavelength to each detector. In the absence of CO and CO_2, the energy received by each detector is maximum. During combustion, the IR absorption properties of CO and CO_2 gases in the chamber cause a loss of energy; therefore a loss in signal results which is proportional to concentrations of each gas in the closed loop. Total carbon, as CO_2 plus CO, is monitored and measured over a period of time. See Figure A.3.

6.4 Infrared absorption for sulfur — Method A

Sulfur dioxide (SO_2) absorbs infrared (IR) energy at a precise wavelength within the IR spectrum. Energy of this wavelength is absorbed as the gas passes through a cell body in which the IR energy is transmitted. All other IR energy is prevented from reaching the detector by use of a precise wavelength filter. Therefore, the absorption of

IR energy can only be attributed to SO_2 and its concentration is measured as changes in energy at the detector. One cell is used as both a reference and a measure chamber. Total sulfur, as SO_2, is monitored and measured over a period of time. See Figure A.4.

6.5 Infrared absorption for sulfur — Method B

During specimen combustion, the flow of SO_2 with its oxygen gas carrier is routed through the measuring chamber (see 5.1) while oxygen alone passes through the reference chamber. Energy from the IR source passes through both chambers, simultaneously arriving at the diaphragm (capacitor plate). Part of the IR energy is absorbed by the SO_2 present in the measuring chamber while none is absorbed passing through the reference chamber. This creates an IR energy imbalance reaching the diaphragm, thus distorting it. This distortion alters the fixed capacitance creating an electric signal change that is amplified for measurement as SO_2. Total SO_2 is monitored and measured over a period of time. See Figure A.5.

6.6 Infrared absorption for sulfur — Method C, closed loop

The combustion is performed in a closed loop where SO_2 is detected in an infrared cell. The SO_2 is measured using a solid state energy detector, and filters are used to pass the appropriate IR wavelength to the detector. During combustion, the IR absorption properties of the SO_2 gas in the chamber causes a loss of energy, therefore a loss in signal results which is proportional to the concentration of the gas in the closed loop. Total sulfur, as SO_2, is monitored and measured over a period of time. See Figure A.6.

7 Sampling

Carry out sampling in accordance with ISO 14284 or appropriate national standards for iron and steel.

8 Procedure

WARNING — The risks related to combustion analysis are mainly burns in pre-igniting the ceramic crucibles and in effecting fusion. 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 effectively removed from the apparatus since a high concentration of oxygen in a confined space can present a fire hazard.

8.1 Preparation of apparatus

Assemble the apparatus and prepare it for operation according to the manufacturer's instructions. Test the furnace and analyser to ensure the absence of leaks. Make a minimum of five determinations using a specimen with measurable concentrations of carbon and sulfur and accelerator as directed in 8.3 before attempting to calibrate the system or determine the blank.

8.2 Test portion

8.2.1 The sample shall be uniform in size, but not finer than 0,4 mm. It shall also be free of oil, grease and other contaminants, particularly those that could augment the carbon and sulfur contents of the sample. The same size of portion of test material shall be taken for both calibration and analysis and shall be in accordance with the manufacturer's instructions.

Wash contaminated samples and those which contain a mass fraction of less than 0,02 % carbon in acetone, cyclohexane or other suitable solvent, and dry at 70 °C to 100 °C.

Weigh, to the nearest 1 mg, a suitable amount of test sample depending on the capability of induction furnace and analyte content.

8.2.2 The laboratory should ensure that its samples are not contaminated with carbon- and/or sulfur-containing materials. Specific sample preparation methods are not included in this International Standard. It is recommended that from time to time the laboratory conduct controlled, replicate determinations on portions of analysis-ready and analysis-ready/solvent-cleaned samples at different concentrations in order to detect contamination. If there are statistically significant differences between the analysis-ready and the analysis-ready/solvent-cleaned portions, sample preparation procedures should be reviewed and revised, as appropriate.

8.3 Calibration

8.3.1 Carbon

8.3.1.1 Establishing calibration ranges and selecting CRMs

Establish the total range of carbon contents to be analysed in the laboratory using this method and then divide it into sub-parts as given in the Table 1.

Table 1 — Calibration range of carbon

Instrument range designation	Calibration range mass fraction %
I	0,005 to 0,120
II	0,10 to 1,25
III	1,0 to 4,3

The instrument ranges given in Table 1 were selected to achieve optimum instrument performance and to set parameters for interlaboratory testing. They overlap slightly to provide operating flexibility. It is important to note that carbon contents with a mass fraction below 0,005 % or above 5 % are outside the tested range and therefore such results cannot be claimed to have been determined in compliance with this standard method. In like manner, laboratories are not obliged to calibrate over all three ranges if these are not required to meet the workload requirements.

Finally, reasonable deviations from these ranges may be made to accommodate to workload requirements the need for flexibility in matching the calibration ranges for both carbon and sulfur in simultaneous determinators.

Select a set of CRMs (4.10) for calibration and verification which, at a minimum, fall at the bottom, top, and quartile points of each operational operating range. In addition, select a CRM very low in carbon content for use in establishing the blank.

8.3.1.2 Adjust response of measurement system

If the instrument has more than one carbon detector (measurement system) perform the adjustment described in this section on each one.

NOTE 1 The sole purpose of this section is to provide a calibration of sufficient accuracy to establish the blank.

Establish all experimental parameters for all ranges of carbon. If simultaneous carbon and sulfur determinations are to be made, make sure that all parameters for the corresponding channels are the same. Parameters to be specified include:

- crucible: to pre-burn or not;
- accelerator: type and mass;
- sample size: mass.

It is highly recommended that crucibles be pre-burned to minimize the magnitude and variability of the blank.

Transfer the selected mass of an RM (4.11) containing about 0,05 % carbon by mass fraction, weighed to the nearest 1 mg and the selected mass of accelerator, weighed to the nearest 5 mg, to a crucible (5.2). Place the crucible on the furnace pedestal and raise the pedestal into position. Use crucible tongs (5.3) to handle pre-burned crucibles.

Start the analysis cycle; referring to the manufacturer's recommended procedure regarding entry of specimen weight and blank value.

Repeat the analysis of the RM (4.11) until the absence of drift is indicated. Following the manufacturer's instructions adjust the signal to provide a reading within 0,003 % mass fraction carbon value for the RM.

8.3.1.3 Determination of blank reading

For each instrument range, transfer the selected mass of accelerator, to the nearest 0,005 g, into a crucible. Add the selected mass of the CRM (4.10) to be used to measure the carbon blank. Enter the mass of the CRM into the instrument's weight compensator. Place the crucible on the furnace pedestal and analyse. Repeat the determination three more times. Average the results. Subtract the carbon content of the CRM from the average to determine the blank value. If the blank is greater than 0,002 % by mass fraction and the standard deviation is greater than 0,000 5 % by mass fraction, find the cause of the problem, fix it, and repeat the experiment. Enter the average blank value into the analyser in accordance with the manufacturer's instructions.

The mass of accelerator taken should be adjusted, if necessary, to match the amount specified in 8.3.1.2. If the analyser does not have automatic blank correction, the blank value should be subtracted from the total result prior to any calculation.

8.3.1.4 Establish calibration curves

Using CRMs (4.10) selected appropriately from the list created in 8.3.1.1, calibrate the instrument for the ranges indicated in 8.3.1.1, using the blank values determined in 8.3.1.3 in accordance with the manufacturer's instructions. If the software supplied with the instrument does not allow curve-fitting with multiple calibrants, calibrate it as instructed by the manufacturer and then immediately analyse all the calibrants as unknowns. Evaluate the results against the manufacturer’s requirements. Correct any non-compliant conditions before continuing to the next step.

8.3.2 Sulfur

8.3.2.1 Establishing calibration range and selecting CRMs

Establish the total range of sulfur contents to be analysed in the laboratory using this method and then divide it into subparts along the following Table 2.

Table 2 — Calibration range of sulfur

Instrument range designation	**Calibration range** mass fraction %
I	0,000 5 to 0,050 0
II	0,03 to 0,33

The instrument ranges given in Table 2 were selected to achieve optimum instrument performance and to set parameters for interlaboratory testing. They overlap slightly to provide operating flexibility. It is important to note that sulfur contents below 0,000 5 % mass fraction or above 0,4 % mass fraction are outside of the tested range and therefore such results cannot be claimed to have been determined in compliance with this standard method. In like manner, laboratories are not obliged to calibrate over both ranges if these are not needed to meet the workload requirements.

Finally, reasonable deviations from these ranges may be made to accommodate to workload requirements the need for flexibility in matching the calibration ranges for both carbon and sulfur in simultaneous determinations.

Select a set of CRMs (4.10) for calibration and verification which, at a minimum, fall at the bottom, top and quartile points of each operational operating range. In addition, select a CRM which is very low in sulfur content for use in establishing the blank.

8.3.2.2 Adjust response of measurement system

Follow the procedure given in 8.3.1.2 but use a reference material containing about 0,03 % mass fraction sulfur.

8.3.2.3 Determination of blank readings

Follow the procedure given in 8.3.1.3, but use an appropriate CRM (4.10) containing sulfur.

8.3.2.4 Establish calibration curves

Using CRMs (4.10) selected appropriately from the list created in 8.3.2.1, calibrate the instrument for the ranges indicated in 8.3.2.1, using the blank values determined in 8.3.2.3, in accordance with the manufacturer's instructions. If the software supplied with the instrument does not allow curve-fitting with multiple calibrants, calibrate it as instructed by the manufacturer and then immediately analyse all of the calibrants as unknowns. Evaluate the results against the manufacturer’s requirements. Correct any non-compliant conditions before continuing to the next step.

8.4 Verification of calibration

8.4.1 Linearity

For each calibration range for carbon and sulfur, pick two CRMs (4.10) which are known to be homogeneous below the 0,1 g sample size level. One should be lower (A), and the other above the upper (B) extremities of the calibration range. Mix these into the nine different blends, analyse them, and assess the linearity of the calibration as described below.

If more than one calibrated range uses the same combination of hardware, including detector, then these ranges may be combined for the linearity test. In such cases samples A and B shall be at the extreme ends of the combined ranges.

Prepare a series of nine crucibles according to the procedure to be used in the laboratory to analyse unknown samples. Using a four-place analytical balance, weigh a portion of each of the RMs (4.11) into each crucible, according to Table 3. Weigh, to the nearest 0,000 1 g, each portion and record the results. In Table 3, M designates the mass of total sample, normally 1 g, to be taken for analysis.

Calculate the concentration of the element to be determined using the formula:

$$c_{\mathrm{E}} = \frac{M_{\mathrm{A}} \times c_{\mathrm{A}} + M_{\mathrm{B}} \times c_{\mathrm{B}}}{M_{\mathrm{A}} + M_{\mathrm{B}}}$$

where

M_{A} is the mass of A in grams;

c_{A} is the concentration of A in grams per cubic centimetre;

M_{B} is the mass of B in grams;

c_{B} is the concentration of B in grams per cubic centimetre.

Analyse the samples in random order without any undue delay and plot the determined values on the Y axis versus the calculated values on the X axis. The plot should be a straight line with a slope of very nearly 45°. If it is not, find the source of the problem, eradicate it, and repeat the experiment.

Calculate the slope of the line defined by crucibles 1 to 3 and the slope of the line defined by crucibles 7 to 9. Subtract the two slopes and calculate the percentage difference between the two slopes. If the percentage difference is greater than 0,2, consider fine-tuning the linearity of the detector or taking other appropriate corrective action and repeating the experiment. If no corrective action is taken at this time, and one or more other performance specifications are not met, then the linearity of the system shall be improved. Record the linearity test data.

An example of a linearity test, with data, is given in annex B.

Table 3 — Series of samples for checking linearity

Sample No.	Mass of A g	Mass of B g
1	M	0
2	$0{,}875 \times M$	$0{,}125 \times M$
3	$0{,}75 \times M$	$0{,}25 \times M$
4	$0{,}625 \times M$	$0{,}375 \times M$
5	$0{,}5 \times M$	$0{,}5 \times M$
6	$0{,}375 \times M$	$0{,}625 \times M$
7	$0{,}25 \times M$	$0{,}75 \times M$
8	$0{,}125 \times M$	$0{,}875 \times M$
9	0	M

8.4.2 Agreement with CRMs

For each range of each element, pick a set of CRMs (4.10) that were not used in either the calibration or the linearity check and that cover at least the low, top and mid-quartile points of the calibration range and analyse them three times each in rapid succession and in random order. Average the found values for each material. Compile the data and obtain the average of the determinations for each CRM and create a table along the following lines:

Instrument range	CRM	Certified value	Found value

Plot the average of the found values versus the certified values and draw a best-fit straight line through the points. Then draw an ideal straight line that would result if all found values were exactly the same as their corresponding certified values. If the differences between these lines (not individual data points) exceed the values in Table 4, recalibrate and take other corrective action as needed to bring the performance of the instrument into this specification. This test ensures that the instrument's calibration is sufficiently bias-free over its calibration range and that its calibration is traceable to a number of reliable CRMs.