
**Steel and iron — Determination of high
sulfur content — Infrared absorption
method after combustion in an induction
furnace**

*Aciers et fontes — Dosage du soufre en fortes teneurs — Méthode par
absorption dans l'infrarouge après combustion dans un four à induction*

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Foreword

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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.

International Standard ISO 13902 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

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

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

1 Scope

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

The method is applicable to a sulfur content of between 0,10 % (*m/m*) and 0,35 % (*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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet— Nominal sizes of openings.*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method.*

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

3 Principle

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 sulfur into sulfur dioxide.

Measurement by infrared absorption of the sulfur dioxide carried by a current of oxygen.

4 Reagents and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

4.1 Oxygen, 99,5 % (*m/m*) minimum.

It is essential that an oxidation catalyst, copper(II) oxide or platinum for example, in a tube heated to a temperature above 450 °C is used prior to a purifying unit (see annex A), when the presence of organic contaminants is suspected in the oxygen.

4.2 Pure iron, of a known (or determined by the procedure given in 7.4) low sulfur content of less than 0,000 5 % (*m/m*).

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

4.4 Magnesium perchlorate, anhydrous, [Mg(ClO₄)₂], particle size: from 0,7 mm to 1,2 mm.

4.5 Accelerator, tungsten, free from sulfur, or of a known sulfur content of less than 0,000 5 % (*m/m*).

NOTE 1 The mesh size of the accelerator may be dependent on the type of apparatus used.

4.6 Anhydrous barium sulfate [minimum assay: 99,5 % (*m/m*)], dried at 105 °C to 110 °C for 3 h and cooled in a desiccator before use.

4.7 Inert ceramic (attapulugus 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.

The apparatus required for combustion in a high-frequency induction furnace and the subsequent infrared absorption measurement of the evolved sulfur dioxide may be obtained commercially from a number of manufacturers. Follow the manufacturer's instructions for the operation of the equipment.

Features of commercial instruments are given in annex A.

5.1 Microbalance, weighing to the nearest 0,001 mg.

5.2 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.

5.3 Glass-fibre filter, cut to the diameter of the crucible and ignited for 12 h at 450 °C.

6 Sampling and preparation of the test samples

Sampling and preparation of the samples shall be carried out in accordance with ISO 14284. The sample for analysis shall be homogenized before weighing the test portion. Powders can be homogenized by stirring (see clause 9).

7 Procedure

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

7.1 Apparatus conditioning

Purify the oxygen supply using tubes packed with the inert ceramic (attapulugus clay) impregnated with sodium hydroxide (4.7) and magnesium perchlorate (4.4), 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 sulfur, adjust the instrument reading for each calibration range as follows.

Select the certified reference material with a sulfur content close to the maximum sulfur content in the calibration series, and measure the sulfur content of the certified reference material in the same manner as specified in 7.4.

Adjust the reading of the instrument to the certified value.

It is essential that this adjustment is 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 (4.3). Evaporate the last traces of the solvent by heating.

Weigh, to the nearest 1 mg, 0,5 g of the test sample (see clause 9) and add 0,5 g \pm 0,001 g of the pure iron (4.2) (see note 2).

NOTE 2 The mass of the test portion and accelerator 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.

Prepare a ceramic crucible (5.2), place a glass-fibre filter (5.3) at the bottom by using a small pair of tweezers. Add 1,000 g of the pure iron (4.2) and 1,5 g \pm 0,1 g of the accelerator (4.5).

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

Obtain the readings of the blank tests and convert them to milligrams of sulfur by means of the calibration graph (7.6).

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

NOTE 3 It is essential that the mean blank value does not exceed 0,005 mg of sulfur and the difference between the two blank values does not exceed 0,003 mg of sulfur. If this value is abnormally high, it should be investigated and the source of contamination eliminated.

7.4 Sulfur content in the pure iron (4.2)

Determine the sulfur content in the pure iron (4.2) as follows.

Prepare two ceramic crucibles(5.2). Place a glass-fibre filter (5.3) at the bottom of each of them by using a small pair of tweezers.

Add 0,500 g of the pure iron (4.2) to one ceramic crucible and 1,000 g to the other. Cover each with $1,5 \text{ g} \pm 0,1 \text{ g}$ of the accelerator (4.5).

Treat the crucible and contents as specified in the second and third paragraphs of 7.5.

Convert the values obtained into milligrams of sulfur by means of the calibration graph (7.6).

The mass (m_2) of sulfur, with 0,500 g of pure iron added, is obtained by subtracting the value (m_3) corresponding to 0,500 g of the pure iron from that (m_4) of 1,000 g of pure iron. The mass (m_5) of sulfur with 1,000 g of pure iron added is twice the mass (m_2) of the sulfur with 0,500 g of pure iron added:

$$m_5 = 2 \times m_2 = 2 \times (m_4 - m_3)$$

7.5 Determination

Prepare a ceramic crucible (5.2) and place a glass-fibre filter (5.3) at the bottom by using a small pair of tweezers. Add the test portion (7.2) and the pure iron (4.2) (see 7.2), and cover with $1,5 \text{ g} \pm 0,1 \text{ g}$ of the accelerator (4.5).

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.

7.6 Establishment of the calibration graph

7.6.1 Preparation of the calibration series

Prepare eight ceramic crucibles (5.2) and place a glass-fibre filter (5.3) at the bottom of each of them by using a small pair of tweezers.

Weigh, with the microbalance (5.1), to the nearest 0,001 mg, the masses of barium sulfate (4.6) as near as possible to those indicated in table 1.

Add 1,000 g of the pure iron (4.2) and cover with $1,5 \text{ g} \pm 0,1 \text{ g}$ of the accelerator (4.5).

7.6.2 Measurements

Treat the crucible and contents as specified in the second and third paragraphs of 7.5.

Table 1 — Calibration series

Mass of barium sulfate (4.6) mg	Mass of sulfur taken in the ceramic crucible mg	Sulfur content in the test portion % (m/m)
0 ¹⁾	0	0
3,64	0,50	0,10
5,46	0,75	0,15
7,28	1,00	0,20
9,10	1,25	0,25
10,92	1,50	0,30
12,74	1,75	0,35
14,56	2,00	0,40

1) Zero member