

SLOVENSKI STANDARD SIST EN 24935:1997

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Jeklo in železo - Določanje žvepla - Infrardeča absorpcijska metoda po zgorevanju v indukcijski peči (ISO 4935:1989)

Steel and iron - Determination of sulphur content - Infrared absorption method after combustion in an induction furnace (ISO 4935:1989)

Stahl und Eisen - Bestimmung des Schwefelgehalts - Methode mit Infrarotabsorption nach Verbrennung im Induktionsofen (ISO 4935-1989) REVIEW

Aciers et fontes - Détermination du soufre - Méthode par absorption dans l'infrarouge apres combustion dans un four a induction (ISO 4935:1989)

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77.080.01 Železne kovine na splošno Ferrous metals in general

SIST EN 24935:1997

en



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English version

Steel and iron - Determination of sulfur content -Infrared absorption method after combustion in an induction furnace (ISO 4935:1989)

Aciers et fontes - Détermination du
soufre - Méthode par absorption dansStahl und Eisen - Bestimmung des
Schwefelgehalts - Methode mitl'infrarouge après combustion dans un
four à induction (ISO 4935:1989)Infrarotabsorption nach Verbrennung im
Induktionsofen (ISO 4935:1989)

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CEN

European Committee for Standardization Comité Européen de Normalisation Europäisches Komitee für Normung

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Ref. No. EN 24935:1991 E

EN 24935:1991

Page 2 EN 24935:1991

Foreword

On the proposal of the Technical Committee ECISS/TC 20 the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization (ECISS) decided in May 1991 to submit the International Standard

ISO 4935:1989 Steel and Iron - Determination of sulfur content - Infrared absorption method after combustion in an induction furnace

to the formal vote.

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This European Standard EN 24935 was approved by CEN on 1991-12-20. (standards.itch.al)

According to the CEN/CENELEC Internal Regulations, the follwing countries are bound to implement this European Standard9:5:1997

https://standards.iteh.ai/catalog/standards/sist/a5763216-d12e-48f2-9c81-Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 4935:1989 was approved by CEN as a European Standard without any modifications.

SIST EN 24935:1997

INTERNATIONAL STANDARD



First edition 1989-09-15

Corrected and reprinted 1990-02-01

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

iTeh STANDARD PREVIEW Méthode par absorption dans l'infrarouge après combustion dans un four à induction

<u>SIST EN 24935:1997</u> https://standards.iteh.ai/catalog/standards/sist/a5763216-d12e-48f2-9c81-605d042a40fa/sist-en-24935-1997



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 VIEW the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting. **Standards.iteh.ai**)

International Standard ISO 4935 was prepared by Technical Committee ISO/TC 17, *Steel.* SIST EN 24935:1997

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Annexes A, B and C of this International Standard are for information and 935-1997

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International Organization for Standardization

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Steel and iron — Determination of sulfur 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 sulfur content in steel and iron.

The method is applicable to sulfur contents between 0,002 % (m/m) and 0,10 % (m/m)

2 Normative references

Reagents and materials

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

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

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

reference in this text, constitute provisions of this International 24935.4.27 Pure iron, of known low sulfur contents less than The following standards contain provisions which, through 0,000,5,% (m/m).48f2-9c81-Standard. At the time of publication date editions tindicated ards/

were valid. All standards are subject to revision (and) parties toist-en-24935-199 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, Laboratoy 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 sulfur into sulfur dioxide.

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

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

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

4.5 Accelerator, tungsten, free of sulfur, or of known sulfur contents less than 0,000 5 % (m/m). The mesh size of the accelerator may be dependent on the type of apparatus used.

4.6 Sulfur, standard solutions.

Weigh, to the nearest 0,1 mg, the mass described in table 1 of potassium sulfate [minimum assay: 99,9 % (m/m)] previously dried at 105 °C to 110 °C for 1 h or to constant mass and cooled in a desiccator.

Table	ə 1
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Reference of sulfur standard solution	sulfate g	of sulfur mg/ml
4.6.1	0,217 4	0,40
4.6.2	0,380 4	0,70
4.6.3	0,543 4	1,00
4.6.4	1,086 9	2,00
4.6.5	1,902 2	3,50
4.6.6	2,717 2	5,00

Transfer to seven 100 ml beakers and dissolve in water.

Transfer quantitatively into seven 100 ml one-mark volumetric flasks. Dilute to the mark with water and mix.

47 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 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 C.

Micropipettes, 50 µl and 100 µl, limit of error shall be 5.1 less than 1 µl. iTeh STAI

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.Standal

Ceramic crucible, capable of withstanding combustion FN 22925-1 Test portion 5.3

in an induction furnace. https://standards.iteh.ai/catalog/standarbegiease7the7test sample by washing in a suitable solvent (4.3). Ignite crucibles in an electric furnace in air or in a current of the vaporate the last traces of the washing liquid by heating.

NOTE -

oxygen for not less than 2 h at 1 100 °C and store in a desiccator before use.

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

6 Sampling

Sampling shall be carried out in accordance with ISO 377 or appropriate national standards for iron.

Procedure 7

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 Apparatus conditioning

Purify the oxygen supply using tubes packed with the inert ceramic (attapulgus clay) impregnated with sodium hydroxide (4.7) and magnesium perchlorate (4.4), and maintain a quies-

cent 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, 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.

specified in 7.5 It cannot replace or correct the calibration.

This adjustment shall be made before the calibration as

Weigh, to the nearest 1 mg, 1 g of the test sample for sulfur contents less than 0,04 % (m/m) and 0,5 g for sulfur contents greater than 0,04 % (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 a tin capsule (5.2) to a ceramic crucible (5.3), press the tin capsule lightly against the bottom of the crucible. Add the same quantity of the pure iron (4.2) as the test portion (7.2) 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 (7.5).

The blank value is obtained by subtracting the mass of sulfur in the pure iron (4.2) used (see note 1) from the mass of sulfur in the blank tests.

The mean blank value (\overline{m}_1) is calculated from the two blank values (see note 2).

NOTES

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

Prepare two ceramic crucibles (5.3). Transfer a tin capsule (5.2) to each and press the capsule lightly against the bottom of the crucible.

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 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.

Convert the values obtained into milligrams of sulfur by means of the calibration graph (7.5).

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 of 1,000 g of pure iron added is twice the mass (m_2) of the sulfur with 0,500 g of pure iron added:

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

2 The mean blank value shall not exceed 0,005 mg of sulfur and the difference between the two blank values shall not exceed 0,003 mg of sulfur. If this value is abnormally high, investigate and eliminate the source of contamination.

7.4 Determination

Transfer a tin capsule (5.2) to a 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 $1,5 g \pm 0,1 g$ of the US accelerator (4.5).

Place the ceramic crucible and contents on the pedestal post, 24935:1997 raise to the combustion position and lock the system: Operate lards/sist/a5763216-d12e-48f2-9c81the furnace in accordance with the manufacturer(s4) instruction structurer(s4) 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 sulfur contents less than 0,005 % (*m*/*m*)

7.5.1.1 Preparation of the calibration series

Using the 50 μ l micropipette (5.1), transfer water (zero member) and sulfur standard solutions (4.6) as indicated in table 2 to four separate tin capsules (5.2).

Evaporate slowly at 90 °C until completely dry and then cool to room temperature in a desiccator.

Ta	b	le	2
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Reference of sulfur standard solution	Mass of sulfur μg	Sulfur content in the test portion % (m/m)
Water	0	0,000 0
4.6.1	20	0.002 0
4.6.2	35	0,003 5
4.6.3	50	0,005 0

7.5.1.2 Measurements

Transfer the tin capsule from 7.5.1.1 to a ceramic crucible (5.3), press the tin capsule lightly against the bottom of the ceramic crucible, add 1,000 g of the pure iron (4.2) and cover with 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.

7.5.1.3 Plotting the calibration graph

The net reading is obtained 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 sulfur for each member of the calibration series.

7.5.2 Samples having sulfur contents between 0,005 % (m/m) and 0,04 % (m/m)

7.5.2.1 Preparation of the calibration series

Using the 50 μ l micropipette (5.1), transfer water (zero member) and sulfur standard solutions (4.6) as indicated in table 3 to five separate tin capsules (5.2).

Evaporate slowly at 90 °C until completely dry and then cool to room temperature in a desiccator.

Reference of sulfur standard solution	Mass of sulfur µg	Sulfur content in the test portion % (m/m)
Water	0	0,000 0
4.6.3	50	0,005 0
4.6.4	100	0,010 0
4.6.6	250	0,025 0
4.6.7	400	0,040 0

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 sulfur contents between 0,04 % (m/m) and 0,1 % (m/m)

7.5.3.1 Preparation of the calibration series

Using the 100 μ l micropipette (5.1), transfer water (zero member) and sulfur standard solutions (4.6) as indicated in table 4 to five separate tin capsules (5.2).