SLOVENSKI STANDARD
SIST EN 27527:2009

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Nikelj, feronikelj in nikljeve zlitine - Doloļ evanje ģvepla - Jodometrijska titracijska metoda po zgorevanju v indukcijski peļ i (ISO 7527:1985)

Nickel, ferronickel and nickel alloys - Determination of sulphur content - Iodometric titration method after induction furnace combustion (ISO 7527:1985)

Nickel, Ferronickel und Nickellegierung - Bestimmung des Schwefelgehaltes - Iodometrisches Titrationsverfahren nach Verbrennung im Induktionsofen (ISO 7527:1985)

Nickel, ferro-nickel et alliages de nickel - Dosage du soufre - Méthode par titration iodométrique après combustion dans un four à induction (ISO 7527:1985)

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Descriptors : Nickel, nickel alloys, ferronickel, chemical analysis, determination of content, sulphur, volumetric method, iodometry, combustion analysis, induction furnaces

English version

Nickel, ferronickel and nickel alloys - Determination of sulfur content - Iodimetric titration method after induction furnace combustion (ISO 7527:1985)

Nickel, ferro-nickel et alliages de nickel - Dosage du soufre - Méthode par titration iodométrique après combustion dans un four à induction (ISO 7527:1985)

Nickel, Ferronickel und Nickellegierung - Bestimmung des Schwefelgehaltes - Iodometrisches Titrationverfahren nach Verbrennung im Induktionsofen (ISO 7527:1985)

This European Standard was approved by CEN on 1991-11-06 and is identical to the ISO standard as referred to. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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CEN

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

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

On the proposal of the CEN Central Secretariat, the Technical Board has decided by resolution BT C17/1990 to submit the International Standard

ISO 7527:1985 : Nickel, ferronickel and nickel alloys - Determination of sulfur content - Iodimetric titration method after induction furnace combustion

to the formal vote.

This European Standard EN 27527 was approved by CEN on 1991-09-24.

According to the CEN/CENELEC Internal Regulations, the following countries are bount to implement this European Standard :

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endoresement notice

The text of the International Standard ISO 7527:1985 was approved by CEN as a European Standard without any modifications.

International Standard 7527

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion

Nickel, ferro-nickel et alliages de nickel — Dosage du soufre — Méthode par titrage iodométrique après combustion dans un four à induction

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Descriptors: nickel, nickel alloys, ferronickel, chemical analysis, determination of content, sulphur, volumetric analysis.

Price based on 7 pages

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.

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 7527 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion

1 Scope and field of application

This International Standard specifies a titrimetric method after combustion for the determination of the sulfur content of nickel and ferronickel in the range 0,001 to 0,3 % (m/m), and of nickel alloys in the range 0,002 to 0,1 % (m/m). Examples of compositions are given in the annex.

2 References

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

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

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

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

ISO 7525, *Nickel — Determination of sulfur content — Methylene blue molecular absorption spectrometric method after generation of hydrogen sulfide.*

3 Principle

Combustion of a test portion in a flow of oxygen at a high temperature in a high frequency induction furnace in the presence of fluxes and accelerators.

Absorption of the sulfur dioxide formed in an acidified starch-iodide solution and continuous titration with potassium iodate standard volumetric solution.

4 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 (O_2), 99,5 % (m/m) minimum.

4.2 Ascarite or **soda lime**, 0,7 to 1,2 mm (14 to 22 mesh).

4.3 Magnesium perchlorate [$Mg(ClO_4)_2$], 0,7 to 1,2 mm (14 to 22 mesh).

4.4 Glass-wool.

4.5 Crucibles and lids.

4.5.1 Ceramic crucibles shall be of precise dimensions so that the sample is positioned correctly in the induction coil of the furnace (see 9.1).

4.5.2 Pre-ignite the crucibles in air or oxygen in a furnace for not less than 1 h at 1 100 °C and store in a desiccator or closed container. A resistance furnace with a combustion tube through which a flow of oxygen passes may be used. Crucible lids, used to help retain the solid oxidation products in the hot zone, are pre-ignited in a similar manner.

4.6 Fluxes: Low sulfur tin, copper plus tin, copper or vanadium pentoxide (see 9.2).

4.7 Accelerators: Low sulfur copper, iron, tungsten or nickel (see 9.2).

4.8 Nickel, low sulfur of known value [< 0,001 % (m/m)].

4.9 Standard reference steel, containing 0,1 to 0,2 % (m/m) sulfur.

4.10 Hydrochloric acid, ϱ_{20} = 1,19 g/ml, diluted 1 + 99.

4.11 Starch-iodide, solution.

Transfer 9 g of soluble starch to a 50 ml beaker, add 5 to 10 ml of water and stir until a smooth paste is obtained. Pour the mixture slowly into 500 ml of boiling water. Cool, add 15 g of potassium iodide and stir until it is dissolved. Dilute to 1 litre with water and mix.

4.12 Potassium iodate, standard volumetric solution.

Dissolve exactly 0,222 5 g of potassium iodate in 900 ml of water containing 1 g of sodium hydroxide. Transfer to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

1 ml of this standard volumetric solution is equivalent to 0,1 mg S.

4.13 Potassium iodate, standard volumetric solution.

Transfer 200 ml of potassium iodate solution (4.12) to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

1 ml of this standard volumetric solution is equivalent to 0,02 mg S.

NOTE — The sulfur equivalents of 4.12 and 4.13 are based on the complete conversion and recovery of sulfur as sulfur dioxide. Well-established standards of known sulfur content are used for solution standardization.

5 Apparatus

5.1 The apparatus required for combustion in a high frequency induction furnace and titration 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 (see 9.3).

5.2 Burettes, of capacity 50 ml, graduated in divisions of 0,1 ml; and of capacity 10 ml, graduated in divisions of 0,02 ml, in accordance with ISO 385/1, class A.

5.3 Pipettes, in accordance with ISO 648, class A.

5.4 Volumetric flasks, in accordance with ISO 1042, class A.

6 Sampling and samples

6.1 Sampling and preparation of the laboratory sample shall be carried out by normal agreed procedures or, in case of dispute, by the relevant International Standard.

6.2 The laboratory sample normally is in the form of a powder, granules, millings or drillings and no further preparation of the sample is necessary.

6.3 If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned by washing with high purity acetone and drying in air.

6.4 If the laboratory sample contains particles or pieces of widely varying sizes, the test portion should be obtained by riffling.

7 Procedure

WARNING — 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.

7.1 Determination

7.1.1 Weigh, to the nearest 0,001 g, 0,9 to 1,1 g of the test sample, and transfer to a pre-ignited crucible (4.5) containing a suitable amount of the preferred flux (4.6). Add the appropriate quantity of accelerator (4.7), if required. The flux and accelerator used will depend on the individual characteristics of the equipment and the type of material being analysed. Typical additions to a 1,0 g test portion are 2 g of copper, 1 g of copper plus 1 g of iron, 2 to 3 g of tungsten, or 1 g of vanadium pentoxide plus 1 g of iron powder. Place the crucible lid in position.

7.1.2 Place the crucible and contents on the pedestal post of the furnace and, with oxygen flowing, raise to the combustion position.

7.1.3 Add 50 to 70 ml of hydrochloric acid (4.10) and 2 ml of starch-iodide solution (4.11) to the absorption vessel. Add sufficient potassium iodate solution (4.13) from a burette to obtain the intensity of the blue colour which will be taken as the end-point of the final titration. Refill the burette to the zero mark.

NOTE — For sulfur contents higher than 0,02 % (*m*/*m*) use the stronger iodate solution (4.12).

7.1.4 Switch on the furnace and combust the sample while passing oxygen through the system. Titrate continuously with the potassium iodate solution (4.13) to maintain the blue starch-iodine colour chosen as the end-point. Do not allow the solution to become colourless at any time during the titration because of possible loss of sulfur dioxide. After the contents of the crucible have combusted completely, as shown by no further decrease of the blue colour, usually after about 5 min, turn off the power supply to the induction coil.

7.1.5 Note the volume of titrant added.

7.1.6 Repeat 7.1.1 to 7.1.5.

7.2 Blank test

7.2.1 Charge a pre-ignited crucible (4.5) with the quantity of flux and accelerator to be used in the determination (7.1) and add 1,00 g of pure nickel of known low sulfur content (4.8).

7.2.2 Proceed as directed in 7.1.2 to 7.1.5.

NOTES

1 The volume of titrant corresponds to the blank due to the crucible, flux, accelerator and sulfur in the pure nickel.

2 The blank should not exceed 0,001 % (*m*/*m*) sulfur.

3 If the blank reading is abnormally high, investigate and eliminate the source of contamination.

7.3 Calibration

7.3.1 Select a certified standard reference steel (4.9).

NOTE — For ferronickel, reference materials with a higher sulfur content are used.

7.3.2 Use the certified standard reference steel in conjunction with pure nickel of low sulfur content [< 0,001 % (m/m)] which is known or has been determined by ISO 7525.

7.3.3 Weigh appropriate proportions of the two materials (7.3.1 and 7.3.2) into a pre-ignited crucible, to cover the high end of the calibration range. Add the preselected amounts of flux and accelerator and combust as directed in 7.1.2 to 7.1.5.

7.3.4 Repeat 7.3.3 to check the repeatability of the reading.

7.3.5 Repeat 7.3.3 with different ratios of the reference sample and pure nickel to provide a calibration check over the required range.

7.3.6 Table 1 illustrates the use of the calibration technique using a certified standard reference steel containing 0,100 % (m/m) S and a reference nickel sample containing 0,001 % (m/m) S.

Table 1 — Calibration example

Mass of steel	Mass of nickel	Sulfur content in composite
g	g	% (m/m)
0,500	0,500	0,050 + 0,000 5
0,300	0,700	0,030 + 0,000 7
0,100	0,900	0,010 + 0,000 9
0	1,000	0,001 0

NOTES

1 It is important that a high temperature be maintained after the sample is fused to ensure complete transfer of the sulfur dioxide from the furnace to the titration vessel.

2 A quiescent combustion is necessary to avoid splashing on to the crucible lid where the fused mass may be removed from the induction heating zone.

7.3.7 Subtract the volume of titrant required for the blank test (7.2) from the volumes of titrant required for each of the calibration standards (7.3.1).

7.3.8 Calculate the mass, in micrograms, of sulfur present in each of the portions of calibration standards combusted and plot against the corresponding volume of titrant corrected for the blank test.

8 Expression of results

8.1 Calculation

8.1.1 Subtract the volume of titrant required for the blank test from the volume required for the test portion.

8.1.2 Read the mass, in micrograms, of sulfur in the test portion from the calibration graph.

8.1.3 The sulfur content, expressed as a percentage by mass, of the test sample is given by the formula

$$\frac{m_1}{m_0} \times 10^4$$

where

m_1 is the mass, in micrograms, of sulfur in the test portion;

m_0 is the mass, in grams, of the test portion.

8.1.4 Report the average of the two results obtained for each test sample.

8.2 Precision

The method specified in this International Standard was subjected to an interlaboratory test programme involving 14 laboratories in six countries. Ten samples were analysed in duplicate, according to the procedure, on two different days.

Repeatability and reproducibility were calculated according to ISO 5725 with the results given in table 2.

9 Notes on procedure and equipment

9.1 Crucibles and lids

Ceramic crucibles are required for containing the sample, any additions which may be necessary and for the subsequent fusion. They shall be of precise dimensions for the system and fit the supporting pedestal post so that the test portion in the crucible is positioned correctly within the induction coil for heating.

Typical dimensions of combustion crucibles are

height	25	mm
external diameter	25	mm
internal diameter	20	mm
wall thickness	2,5	mm
thickness of base	8	mm

Crucibles are pre-ignited at 1 100 °C in oxygen to remove sulfur. Lids, placed on the crucible, help to retain the solid oxidation products in the hot zone of the induction coil. The crucible lids are pre-ignited in a similar manner to the crucibles (see 4.5.2).