SLOVENSKI STANDARD
SIST EN 27526:2009

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Nikelj, feronikelj in nikljeve zlitine - Določ evanje ǵvepla - Metoda infrardeč e absorpcije po zgorevanju v indukcijski peč i (ISO 7526:1985)

Nickel, ferronickel and nickel alloys - Determination of sulphur content - Infra-red absorption method after induction furnace combustion (ISO 7526:1985)

Nickel, Ferronickel und Nickellegierungen - Bestimmung des Schwefelgehaltes - Verfahren der Infrarotabsorption nach Verbrennung im Induktionsofen (ISO 7526:1985)

Nickel, ferro-nickel et alliages de nickel - Dosage du soufre - Méthode par absorption dans l'infrarouge après combustion dans un four à induction (ISO 7526:1985)

Ta slovenski standard je istoveten z: **EN 27526:1991**

ICS:

77.120.40	Nikelj, krom in njune zlitine	Nickel, chromium and their alloys

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EUROPEAN STANDARD

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UDC 669.24:669.15'24-198:620.1:543.422.4:543.845

Descriptors : Nickel, nickel alloys, ferronickel, chemical analysis, determination of content, sulphur, infrared spectroscopy, combustion analysis, induction furnaces

English version

Nickel, ferronickel and nickel alloys - Determination of sulfur content - Infra-red absorption method after induction furnace combustion (ISO 7526:1985)

Nickel, ferro-nickel et alliages de nickel - Dosage du soufre - Méthode par absorption dans l'infrarouge après combustion dans un four à induction (ISO 7526:1985)

Nickel, Ferronickel und Nickellegierungen - Bestimmung des Schwefelgehaltes - Verfahren der Infrarotabsorption nach Verbrennung im Induktionsofen (ISO 7526:1985)

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.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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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 7526:1985 : Nickel, ferronickel and nickel alloys - Determination of sulfur content - Infra-red absorption method after induction furnace combustion

to the formal vote.

This European Standard EN 27526 was approved by CEN on 1991-09-24.

According to the CEN/CENELEC Internal Regulations, the follwing 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 7526:1985 was approved by CEN as a European Standard without any modifications.

International Standard 7526

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

Nickel, ferronickel and nickel alloys — Determination of sulfur content — Infra-red absorption method after induction furnace combustion

Nickel, ferro-nickel et alliages de nickel — Dosage du soufre — Méthode par absorption dans l'infrarouge après combustion dans un four à induction

First edition — 1985-12-15

UDC 669.24 : 543.422.4 : 543.845 **Ref. No. ISO 7526-1985 (E)**

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

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 7526 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 — Infra-red absorption method after induction furnace combustion

1 Scope and field of application

This International Standard specifies an infra-red absorption 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 0,001 to 0,1 % (*m/m*). Examples of compositions are given in annex A.

NOTE — It may be possible to apply this method in the range 0,000 2 to 0,001 % (*m/m*). However, there were insufficient laboratory test data to support the inclusion of this lower level in the scope.

2 References

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.

Measurement of the sulfur dioxide formed using an infra-red analyser and an integration procedure.

4 Reagents and materials

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 steels, containing 0,1 to 0,2 % (*m/m*) sulfur.

5 Apparatus

The apparatus required for combustion in a high frequency induction furnace and the subsequent infra-red 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. A pressure regulator is required to control the oxygen pressure to the furnace according to the manufacturer's specification (usually 28 kN/m^2). Features of commercial equipment are given in annex B.

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. 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 Stabilizing the equipment

7.1.1 Condition and stabilize the equipment by combusting several samples, similar to those to be analysed (7.4), using appropriate fluxes and accelerators.

NOTE — It is not necessary to use pre-ignited crucibles.

7.1.2 Allow the instrument to cycle several times with oxygen flowing and adjust the instrument zero.

7.2 Blank test and zero adjustment

7.2.1 Charge a pre-ignited crucible (4.5) with the quantity of flux and accelerator to be used in the determination (7.4) and add 1,00 g of pure nickel of known low sulfur content (4.8).

7.2.2 Place the crucible and contents on the pedestal post of the furnace, raise to the combustion position and lock the system. Operate the furnace in accordance with the manufacturer's instructions. See 9.3 and annex B.

NOTES

1 The reading obtained 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.

7.2.3 Adjust the instrument reading using the zero adjust or, on some instruments, the blank offset control, to read the sulfur value of the nickel (4.8).

7.2.4 Repeat 7.2.1 to 7.2.3 to obtain a reproducible reading within the precision limits of the instrument.

NOTE — An alternative procedure is to record the reading of the blank test and make the correction using a calibration graph.

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 in 7.2.2. Note the instrument reading.

7.3.4 Adjust the instrument reading to correspond to the correct level of sulfur in the mixture (7.3.3) according to the manufacturer's operating instructions.

7.3.5 Repeat 7.3.3 to check the repeatability of the reading.

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

7.3.7 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

7.4 Determination

7.4.1 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.

7.4.2 Place the crucible and contents on the pedestal post of the furnace, raise to the combustion position and lock the system. Operate the furnace in accordance with the manufacturer's instructions. See 9.3 and annex B.

7.4.3 Record the analyser reading and repeat the determination.

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 infra-red analyser.

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.

8 Expression of results

8.1 Calculation

8.1.1 If the instrument has been calibrated to give a read-out directly as a percentage by mass of sulfur with automatic compensation for the mass of the test portion, take the average of the two determinations and report the result.

8.1.2 If the instrument has been calibrated based on a 1,00 g test portion and does not have automatic mass compensation, divide each reading by the respective mass, in grams, of the test portion. Average the two determinations and report the result.

8.1.3 With some instruments it will be necessary to prepare a calibration graph of instrument reading versus the mass, in micrograms, of sulfur. Read off the graph the mass, in micrograms, of sulfur in the test portion, correct for the blank and mass of the test portion. Average the two determinations and report the result.

8.2 Precision

The method specified in this International Standard was subjected to an interlaboratory test programme involving 14 laboratories in six countries. Eleven 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.

9.2 Fluxes and accelerators

9.2.1 A flux addition has the effect of bonding together small particles of sample for more effective furnace coupling and to produce a more fluid melt. Tin, copper plus tin, copper and vanadium pentoxide have been found satisfactory.

Table 2 — Results of statistical analysis

Metal or alloy	Mean sulfur content [% (*m*/*m*)]	Within laboratory standard deviation, s_w	Between laboratories standard deviation, s_b	Repeatability, r	Reproducibility, R
Ferronickel					
A22	0,020	0,000 6	0,001 3	0,001 8	0,004 0
A28	0,023	0,000 3	0,001 7	0,000 9	0,005 0
C1	0,024	0,000 5	0,001 0	0,001 4	0,003 2
C2	0,048	0,000 7	0,003 0	0,002 1	0,008 9
C3	0,074	0,000 9	0,003 1	0,002 5	0,009 1
C4	0,20	0,003 3	0,013	0,009	0,038
Nickel					
HN*	0,000 4	0,000 14	0,000 15	0,000 4	0,000 5
YG	0,006 3	0,000 2	0,000 5	0,000 5	0,001 6
YF	0,013	0,000 3	0,001 3	0,000 7	0,003 8
Nickel alloy					
AK (H**)	0,002 5	0,000 2	0,000 6	0,000 6	0,001 9
AO (B**)	0,016	0,000 6	0,001 0	0,001 8	0,003 3

* Below scope, included for information.

** Refers to alloy type in table 5.