



SLOVENSKI STANDARD
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Jeklene in železove litine - Določevanje niklja - Plamenska atomska absorpcijska spektrometrična metoda (FAAS)

Steels and cast irons - Determination of nickel content - Flame atomic absorption spectrometric method (FAAS)

Stahl und Eisen Stahl und Eisen - Bestimmung des Nickelanteils -
Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Aciers et fontes - Détermination de la teneur en nickel - Méthode par spectrométrie
d'absorption atomique dans la flamme (SAAF)

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Steels and cast irons - Determination of nickel content - Flame atomic absorption spectrometric method (FAAS)

Aciers et fontes - Détermination de la teneur en nickel -
Méthode par spectrométrie d'absorption atomique
dans la flamme (SAAF)

Stahl und Eisen Stahl und Eisen - Bestimmung des
Nickelanteils -
Flammenatomabsorptionsspektrometrisches
Verfahren (FAAS)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 10136:2018) has been prepared by Technical Committee ECISS/TC 102 “Methods of chemical analysis for iron and steel”, the secretariat of which is held by SIS.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 10136:1989.

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prEN 10136:2018 (E)

1 Scope

This document specifies a flame atomic absorption spectrometric method (FAAS) for the determination of nickel content in steels and cast irons.

The method is applicable to nickel contents between 0,004 % and 2,0 %.

The method can be adapted to lower or higher nickel contents by changing the test portion or the dilution process, provided the criteria in 6.2.2 and 6.2.3 are still met.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 648, *Laboratory glassware - Single-volume pipettes (ISO 648)*

EN ISO 1042, *Laboratory glassware - One-mark volumetric flasks (ISO 1042)*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principle

Dissolution of a test portion in a mixture of appropriate acids and fuming with perchloric acid.

Nebulisation of the test solution into an air/acetylene flame of an atomic absorption spectrometer.

Spectrometric measurement of the atomic absorption of the 232,0 nm or 352,5 nm spectral line emitted by a nickel hollow-cathode lamp.

NOTE Other suitable radiation sources can also be used, provided the criteria in 6.2.2 and 6.2.3 are still met.

5 Reagents

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

The following concentrations and amounts can be modified, provided the changes are taken into account in 8.3 and Clause 9.

5.1 Pure iron, with nickel content < 0,000 5 %.

5.2 Hydrochloric-nitric acids mixture.

Mix three volumes of hydrochloric acid ($\rho_{20} = 1,19$ g/ml, approximately), one volume of nitric acid ($\rho_{20} = 1,40$ g/ml, approximately) and two volumes of water.

This mixture shall be prepared immediately before use.

5.3 Nitric-perchloric acids mixture.

Mix 100 ml of nitric acid ($\rho_{20} = 1,40$ g/ml, approximately) with 800 ml of perchloric acid ($\rho_{20} = 1,54$ g/ml, approximately). Dilute to one litre with water and mix.

Perchloric acid ($\rho_{20} = 1,67$ g/ml approximately) may also be used. 100 ml of perchloric acid (ρ_{20} approximately 1,54 g/ml) are equivalent to 79 ml of perchloric acid ($\rho_{20} = 1,67$ g/ml approximately).

5.4 Nickel standard solution, 1 g/l.

Weigh ($0,500 \pm 0,001$) g of nickel ($\text{Ni} \geq 99,9\%$) and transfer into a heat-resistant glassware of suitable size. Add 25 ml of nitric acid ($\rho_{20} = 1,40$ g/ml, approximately), diluted 1 + 1. Cover with a watch glass and, if necessary, heat gently to assist dissolution. When dissolution is complete boil to remove nitrogen oxides. Allow to cool to room temperature and transfer the solution quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1,0 mg of nickel.

5.5 Nickel standard solution, 0,04 g/l.

Transfer 10,0 ml of nickel standard solution (5.4) into a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,04 mg of nickel.

5.6 Iron base solution, 40 g/l.

Weigh, to the nearest 0,01 g, 10,00 g of pure iron (5.1) and transfer into a heat-resistant glassware of suitable size. Add 100 ml of the hydrochloric-nitric acids mixture (5.2), cover the beaker with a watch glass and heat gently until the iron is dissolved. Add 150 ml of the nitric-perchloric acids mixture (5.3) and heat until dense white fumes of perchloric acid appear. Continue heating for one minute and allow to cool.

In order to get a better mastery of the acidity (volume of free perchloric acid) of the sample solutions and of the calibration solutions, perchloric fumes may be maintained until iron crystallizes.

Add 100 ml of water and heat gently to dissolve the salts. Cool again and transfer the solution quantitatively into a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix.

5.7 Iron base solution, 2 g/l.

Transfer 10,0 ml of iron base solution (5.6) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix.

6 Apparatus

6.1 Ordinary laboratory apparatus

All volumetric glassware shall be class A, in accordance with EN ISO 648 or EN ISO 1042, as appropriate.

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6.2 Atomic absorption spectrometer

6.2.1 General

The spectrometer shall be equipped with a nickel hollow-cathode lamp or other suitable radiation source and supplied with air and acetylene sufficiently pure to give a steady clear fuel-lean flame, free from water and oil, and free from nickel.

The atomic absorption spectrometer used will be satisfactory if, after optimization according to 8.3.4, the limit of detection and characteristic concentration are in reasonable agreement with the values given by the manufacturer and it meets the performance criteria given in 6.2.2 and 6.2.3.

6.2.2 Minimum precision

The standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution shall not exceed 1,0 % of the mean absorbance of this solution.

The standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution (excluding the zero member) shall not exceed 0,5 % of the mean absorbance of the most concentrated calibration solution.

6.2.3 Additional performance requirements

6.2.3.1 General

It is also desirable that the instrument should conform to the additional performance requirements given in 6.2.3.2 and 6.2.3.3.

6.2.3.2 Characteristic concentration

The characteristic concentration for nickel in a matrix similar to the final test solution shall be lower than 0,10 µg/ml.

6.2.3.3 Limit of detection

The limit of detection is a number, expressed in units of concentration (or amount) that describes the lowest concentration level (or amount) of an element that can be determined to be statistically different from an analytical blank.

The limit of detection of nickel in a matrix similar to the final test solution shall be less than 0,15 µg/ml of nickel.

7 Sampling

Carry out sampling in accordance with EN ISO 14284 or appropriate national standards for steels and cast irons.

8 Procedure

WARNING — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic matter in general. All evaporations shall be carried out in fume cupboards suitable for use with perchloric acid.

NOTE The following concentrations, amounts and glassware volumes can be modified, provided the changes are taken into account in all appropriate subclauses of Clauses 8 and 9.

8.1 Test portion

Weigh to the nearest 1 mg, approximately 1,0 g of the test sample.

8.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all reagents as used for the determination.

8.3 Determination

8.3.1 Preparation of the test solution

Transfer the test portion (8.1) into a 250 ml beaker. Add 10 ml of hydrochloric-nitric acids mixture (5.2), cover the beaker with a watch glass and heat gently until the acid action ceases.

NOTE If the test portion is readily soluble in the nitric-perchloric acids mixture (5.3), the addition of the hydrochloric-nitric acids mixture (5.2) can be omitted. In that case, the test portion is dissolved in the nitric-perchloric acids mixture (5.3) and the solution thus obtained is heated as described below.

Add 15 ml of nitric-perchloric acids mixture (5.3) and heat until dense white fumes of perchloric acid appear. Continue heating for one minute and allow to cool.

In order to ensure that the acid content of the sample solutions and of the calibration solutions is the same, the solution should be fumed until crystallisation occurs.

8.3.2 Treatment of the test solution

8.3.2.1 Nickel contents up to 0,10 %

Add 25 ml of water and heat gently to dissolve the salts. Cool again and transfer the test solution quantitatively into a 100 ml one-mark volumetric flask.

Dilute to the mark with water and mix.

If some residue has been left in the solution due to silicon, tungsten, niobium or tantalum, filter the solution through a dry, medium-texture filter paper and collect the filtrate in a dry beaker, after discarding the first portions.

8.3.2.2 Nickel contents between 0,08 % and 2,0 %

Transfer 10,0 ml of the filtrate (8.3.2.1) into a 200 ml one-mark volumetric flask, dilute to the mark with water and mix.

If the test solution has to be diluted, the blank test solution shall be diluted in the same way.

8.3.3 Preparation of the calibration solutions

8.3.3.1 General

Table 1 and Table 2 contain typical sets of calibration solutions. The range of concentrations in a set of solutions may be changed in order to better bracket the expected sample solution concentration.

8.3.3.2 Nickel contents up to 0,10 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of nickel standard solution (5.5) shown in Table 1 and 25 ml of the iron base solution (5.6). Dilute to the mark with water and mix.

Table 1 — Calibration for nickel contents up to 0,1 %

Nickel standard solution volume (5.5) ml	Corresponding nickel mass mg	Corresponding nickel concentration after final dilution mg/ml	Corresponding nickel content in the sample %
0	0	0	0
1,0	0,04	0,000 4	0,004
2,0	0,08	0,000 8	0,008
2,5	0,10	0,001	0,010
5,0	0,20	0,002	0,020
10,0	0,40	0,004	0,040
15,0	0,60	0,006	0,060
20,0	0,80	0,008	0,080
25,0	1,00	0,010	0,10

8.3.3.3 Nickel contents between 0,08 % and 2,0 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of nickel standard solution (5.5) shown in Table 2 and 25 ml of the iron base solution (5.7). Dilute to the mark with water and mix.

Table 2 — Calibration for nickel contents between 0,08 % and 2,0 %

Nickel standard solution volume (5.5) ml	Corresponding nickel mass mg	Corresponding nickel concentration after final dilution mg/ml	Corresponding nickel content in the sample %
0	0	0	0
1,0	0,04	0,000 4	0,08
2,5	0,10	0,001	0,20
5,0	0,20	0,002	0,40
10,0	0,40	0,004	0,80
15,0	0,60	0,006	1,20
20,0	0,80	0,008	1,60
25,0	1,00	0,010	2,00

8.3.4 Adjustment of the atomic absorption spectrometer

Fit the nickel hollow-cathode lamp (see 6.2) to the atomic absorption spectrometer (6.2), switch on the current and allow it to stabilize. Adjust the wavelength in the region of 232,0 nm to minimum absorbance, if possible. Following manufacturer's instructions, fit the correct burner, light the flame and allow the burner temperature to stabilize. Taking careful note of the manufacturer's instructions regarding the minimum flow rate of acetylene, aspirate the calibration solution of highest concentration of analyte and adjust the burner configuration and gas flows to obtain maximum absorbance.

NOTE 1 Other suitable radiation sources can also be used.