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Legerat stål – Bestämning av nickelhalt – Metod med optisk emissionsspektrometri med induktivt kopplat plasma

Alloyed steels – Determination of nickel content – Inductively coupled plasma optical emission spectrometric method

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EUROPEAN STANDARD

EN 10361

NORME EUROPÉENNE

EUROPÄISCHE NORM

December 2015

ICS 77.040.30

English Version

**Alloyed steels - Determination of nickel content -
Inductively coupled plasma optical emission spectrometric
method**

Aciers alliés - Détermination du nickel - Méthode par
spectrométrie d'émission optique avec source à plasma
induit

Legierte Stähle - Bestimmung des Nickelanteils -
Verfahren mittels optischer Emissionsspektrometrie
mit induktiv gekoppeltem Plasma

This European Standard was approved by CEN on 20 June 2015.

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents		Page
European foreword		3
1	Scope	4
2	Normative references	4
3	Principle	4
4	Reagents	4
5	Apparatus	5
6	Sampling	6
7	Procedure	6
8	Determination	8
9	Expression of the results	9
10	Test report	9
Annex A (informative) Plasma optical emission spectrometer - Suggested performance criteria to be checked		13
Annex B (informative) Composition of the samples used for the validation precision test		15
Annex C (informative) Graphical representation of the precision data		16
Bibliography		17

European foreword

This document (EN 10361:2015) has been prepared by Technical Committee ECISS/TC 102 “Methods of chemical analysis of iron and steel”, the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2016 and conflicting national standards shall be withdrawn at the latest by June 2016.

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1 Scope

This European Standard specifies an inductively coupled plasma optical emission spectrometric method for the determination of nickel content (mass fraction) between 5,0 % and 25,0 % in alloyed steels.

The method does not apply to alloyed steels having niobium and/or tungsten contents higher than 0,1 %.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 Principle

Dissolution of a test portion with hydrochloric and nitric acids. Filtration and ignition of the acid insoluble residue. Removal of silica with hydrofluoric acid. Fusion of the residue with potassium hydrogen sulphate (or with potassium disulphate), dissolution of the melt with acid and addition of this solution to the reserved filtrate.

After suitable dilution and, if necessary, addition of an internal reference element, nebulization of the solution into an inductively coupled plasma emission spectrometer and measurement of the intensity of the emitted light (including, where appropriate, that of the internal reference element).

The method uses a calibration based on a very close matrix matching of the calibration solutions to the sample and bracketing of the mass fractions between 0,95 to 1,05 of the approximate content of nickel in the sample to be analysed. The content of all elements in the sample has, therefore, to be approximately known. If the contents are not known the sample shall be analysed by some semi quantitative method. The advantage with this procedure is that all possible interferences from the matrix will be compensated, which will result in high accuracy. This is most important for spectral interferences, which can be severe in very highly alloyed matrixes. All possible interferences shall be kept at a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria specified in the method for the selected analytical lines.

The optical lines reported in the Table 1 have been investigated and the strongest possible interferences are given. If other optical lines are used, they shall be carefully checked. The analytical line for the internal reference element should be selected carefully. The use of scandium at 363,1 nm or yttrium at 371,0 nm is recommended. These lines are interference-free for the elements and contents generally found in alloyed steels.

4 Reagents

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

The same reagents should be used for the preparation of calibration solutions and of sample solutions.

4.1 Hydrochloric acid, HCl ($\rho_{20} = 1,19$ g/ml).

4.2 Nitric acid, HNO₃ ($\rho_{20} = 1,33$ g/ml).

4.3 Hydrofluoric acid, HF ($\rho_{20} = 1,13$ g/ml).

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate, and seek immediate medical treatment.

4.4 Sulphuric acid, H₂SO₄ ($\rho_{20} = 1,84$ g/ml).

4.5 Sulphuric acid, solution 1 + 1.

While cooling, add 25 ml of sulphuric acid (4.4) to 25 ml of water.

4.6 Potassium hydrogen sulphate [KHSO₄] or potassium disulphate [K₂S₂O₇].

4.7 Nickel standard solution, 10 g/l.

Weigh, to the nearest 0,001 g, 5 g of high purity nickel [min 99,9 % (mass fraction)], place it in a beaker and dissolve in 50 ml of water and 100 ml of nitric acid (4.2). Cover with a watch glass and heat gently until the nickel is completely dissolved. Cool and transfer quantitatively into a 500 ml one-mark volumetric flask. Dilute to mark with water and mix.

1 ml of this solution contains 10 mg of nickel.

4.8 Nickel standard solution, 5 g/l.

Weigh, to the nearest 0,001 g, 2,5 g of high purity nickel [min 99,9 % (mass fraction)], place it in a beaker and dissolve in 25 ml of water and 50 ml of nitric acid (4.2). Cover with a watch glass and heat gently until the nickel is completely dissolved. Cool and transfer quantitatively into a 500 ml one-mark volumetric flask. Dilute to mark with water and mix.

1 ml of this solution contains 5 mg of nickel.

4.9 Standard solutions of matrix elements.

Prepare standard solutions for each element whose mass fraction is higher than 1 % in the test sample. Use pure metals or chemical substances with nickel mass fractions less than 100 µg/g.

4.10 Internal reference element solution, 1 g/l.

Choose a suitable element to be added as internal reference and prepare a 1 g/l solution.

NOTE Elements such as In, Sc and Y were used during the precision test of this method.

5 Apparatus

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

5.1 Medium texture filter paper.

5.2 Platinum crucibles.

5.3 Optical emission spectrometer, equipped with inductively coupled plasma.

This shall be equipped with a nebulization system. The instrument used will be satisfactory if, after optimizing in accordance with the manufacturer's instructions, it meets the performance criteria given in Annex A.

The spectrometer can be either a simultaneous or a sequential one. If a sequential spectrometer can be equipped with an extra arrangement for simultaneous measurement of the internal reference element line, it can be used with the internal reference method. If the sequential spectrometer is not equipped with this arrangement, an internal reference cannot be used and an alternative measurement technique without internal reference element shall be used.

6 Sampling

Sampling shall be carried out in accordance with EN ISO 14284 or with an appropriate national standard for steels.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the test sample.

7.2 Preparation of the test solution, T_{Ni}

Transfer the test portion (7.1) into a 250 ml beaker.

Add 15 ml of hydrochloric acid (4.1), cover with a watch glass, heat gently until the attack reaction ceases, and then add dropwise, 10 ml of nitric acid (4.2).

Depending on the composition of each sample, larger amounts of hydrochloric acid may be necessary. Addition of hydrogen peroxide (H_2O_2) may advantageously help dissolution. The same quantities of the dissolution reagents shall be added to the corresponding calibration solutions.

Boil until nitrous fumes have been expelled. After cooling, add about 20 ml of water, filter the solution through a medium texture filter paper (5.1) and collect the filtrate into a 200 ml one-mark volumetric flask.

Wash the filter paper and its content with warm water slightly acidified with nitric acid (4.2) several times and collect the washings in the 200 ml one-mark volumetric flask.

Transfer the filter into a platinum crucible (5.2), dry and ignite first at a relatively low temperature (until all carbonaceous matter is removed) and then at about 800 °C for at least 15 min.

Allow the crucible to cool. Add into the crucible 0,5 ml to 1,0 ml of sulphuric acid solution (4.5) and 2 ml of hydrofluoric acid (4.3). Evaporate to dryness and cool.

Add into the crucible 1,00 g of potassium hydrogen sulphate or potassium disulphate (4.6) and fuse carefully by means of a Meker burner, until a clear melt is obtained.

NOTE 1 For residues containing substantial amounts of chromium carbides, prolonged heating may be necessary for complete fusion. The potassium hydrogen sulphate can be regenerated by allowing the melt to cool, adding some drops of sulphuric acid (4.4) and repeating the fusion until the residue is fused.

NOTE 2 Depending on the composition of each sample, larger amounts of potassium hydrogen sulphate or potassium disulphate (4.6) can be used, provided the same amount is added to the corresponding calibration solutions.

Allow the crucible to cool and add about 10 ml of water and 2 ml of hydrochloric acid (4.1) to the solidified melt. Heat gently, in order to dissolve the fusion products. Allow the crucible to cool and transfer the solution quantitatively to the filtrate in the 200 ml one-mark volumetric flask.

NOTE 3 The volume of hydrochloric acid (4.1) can be increased, provided the same volume is added to the appropriate calibration solutions.

Dilute to the mark with water and mix.

Transfer 20 ml of this sample solution into a 100 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.1).

NOTE 4 Depending on the instrument performances, the final concentration of the test solution may be lower (or higher), provided the corresponding calibration solutions have the same final concentration.

If an internal reference element is used add, with a calibrated pipette, 10 ml of the internal reference element solution (4.10).

NOTE 5 Depending on the instrument performances, the volume and/or the concentration of the internal reference element solution may be different.

Dilute to the mark with water and mix.

7.3 Predetermination of the test solution

Prepare two calibration solutions labelled K_{25} and K_0 , matrix matched to the test sample solution as follows:

Add 25 ml of the nickel standard solution (4.7) in a 400 ml beaker, labelled K_{25} .

In each 400 ml beaker, K_{25} and K_0 , add the volumes of the standard solutions (4.9) necessary to match the sample matrix to be tested, for each element whose content is above 1 %.

The matrix shall be matched to the nearest percent.

Add in each 400 ml beaker, 15 ml of hydrochloric acid (4.1) and 10 ml of nitric acid (4.2). Cover with a watch glass and boil until nitrous fumes have been expelled and, if necessary, until the volume of the solutions is sufficiently reduced. After cooling, add about 20 ml of water and transfer each solution into a 200 ml one-mark volumetric flask.

Dissolve into each flask 1,00 g of potassium hydrogen sulphate or potassium disulphate (4.6) and add 2 ml of hydrochloric acid (4.1).

Dilute to the mark with water and mix.

Transfer 20 ml of each solution K_{25} and K_0 into two 100 ml one-mark volumetric flasks and add 10 ml of hydrochloric acid (4.1).

If an internal reference element is used add 10 ml of the internal reference element solution (4.10).

NOTE Depending on the instrument performances, the volume and/or the concentration of the internal reference element solution may be different.

Dilute to the mark with water and mix.

Measure the absolute intensities I_{25} and I_0 for the solutions K_{25} and K_0 .

Measure the absolute intensity I_{Ni} of the solution of test T_{Ni} .

Calculate the approximate concentration of nickel K_{Ni} in % (mass fraction), in the test solution using Formula (1):

$$K_{Ni} (\%) = I_{Ni} \frac{(K_{25} - K_0)}{I_{25} - I_0} \quad (1)$$

7.4 Preparation of calibration solutions for bracketing: $T_{l,Ni}$ and $T_{h,Ni}$

For each test solution T_{Ni} prepare two matrix matched calibration solutions, $T_{l,Ni}$ and $T_{h,Ni}$ with nickel concentrations in $T_{l,Ni}$ slightly below and in $T_{h,Ni}$ slightly above the concentration in the test solution as follows: