

SVENSK STANDARD

SS-EN 10361:2015

Fastställt/Approved: 2015-12-15
Publicerad/Published: 2015-12-22
Utgåva/Edition: 1
Språk/Language: engelska/English
ICS: 77.040.30

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

This preview is downloaded from www.sis.se. Buy the entire standard via <https://www.sis.se/std-8017881>

Standarder får världen att fungera

SIS (Swedish Standards Institute) är en fristående ideell förening med medlemmar från både privat och offentlig sektor. Vi är en del av det europeiska och globala nätverk som utarbetar internationella standarder. Standarder är dokumenterad kunskap utvecklad av framstående aktörer inom industri, näringsliv och samhälle och befrämjar handel över gränser, bidrar till att processer och produkter blir säkrare samt effektiviserar din verksamhet.

Delta och påverka

Som medlem i SIS har du möjlighet att påverka framtida standarder inom ditt område på nationell, europeisk och global nivå. Du får samtidigt tillgång till tidig information om utvecklingen inom din bransch.

Ta del av det färdiga arbetet

Vi erbjuder våra kunder allt som rör standarder och deras tillämpning. Hos oss kan du köpa alla publikationer du behöver – allt från enskilda standarder, tekniska rapporter och standardpaket till handböcker och onlinetjänster. Genom vår webbtjänst e-nav får du tillgång till ett lättnavigerat bibliotek där alla standarder som är aktuella för ditt företag finns tillgängliga. Standarder och handböcker är källor till kunskap. Vi säljer dem.

Utveckla din kompetens och lyckas bättre i ditt arbete

Hos SIS kan du gå öppna eller företagsinterna utbildningar kring innehåll och tillämpning av standarder. Genom vår närhet till den internationella utvecklingen och ISO får du rätt kunskap i rätt tid, direkt från källan. Med vår kunskap om standarders möjligheter hjälper vi våra kunder att skapa verklig nytta och lönsamhet i sina verksamheter.

Vill du veta mer om SIS eller hur standarder kan effektivisera din verksamhet är du välkommen in på www.sis.se eller ta kontakt med oss på tel 08-555 523 00.



Standards make the world go round

SIS (Swedish Standards Institute) is an independent non-profit organisation with members from both the private and public sectors. We are part of the European and global network that draws up international standards. Standards consist of documented knowledge developed by prominent actors within the industry, business world and society. They promote cross-border trade, they help to make processes and products safer and they streamline your organisation.

Take part and have influence

As a member of SIS you will have the possibility to participate in standardization activities on national, European and global level. The membership in SIS will give you the opportunity to influence future standards and gain access to early stage information about developments within your field.

Get to know the finished work

We offer our customers everything in connection with standards and their application. You can purchase all the publications you need from us - everything from individual standards, technical reports and standard packages through to manuals and online services. Our web service e-nav gives you access to an easy-to-navigate library where all standards that are relevant to your company are available. Standards and manuals are sources of knowledge. We sell them.

Increase understanding and improve perception

With SIS you can undergo either shared or in-house training in the content and application of standards. Thanks to our proximity to international development and ISO you receive the right knowledge at the right time, direct from the source. With our knowledge about the potential of standards, we assist our customers in creating tangible benefit and profitability in their organisations.

If you want to know more about SIS, or how standards can streamline your organisation, please visit www.sis.se or contact us on phone +46 (0)8-555 523 00



Europastandarden EN 10361:2015 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN 10361:2015.

The European Standard EN 10361:2015 has the status of a Swedish Standard. This document contains the official English version of EN 10361:2015.

© Copyright/Upphovsrätten till denna produkt tillhör SIS, Swedish Standards Institute, Stockholm, Sverige. Användningen av denna produkt regleras av slutanvändarlicensen som återfinns i denna produkt, se standardens sista sidor.

© Copyright SIS, Swedish Standards Institute, Stockholm, Sweden. All rights reserved. The use of this product is governed by the end-user licence for this product. You will find the licence in the end of this document.

Uppllysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna uppllysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Kemiska analysmetoder för metaller, SIS/TK 122.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

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.

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 CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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: