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BSI Standards Publication

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

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National foreword

This British Standard is the UK implementation of EN 10361:2015.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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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

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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of an identical text or by endorsement, at the latest by June 2016 and conflicting national standards shall be withdrawn at the latest by June 2016.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 (ρ_{20} = 1,19 g/ml).

4.2 Nitric acid, HNO_3 (ρ_{20} = 1,33 g/ml).

4.3 Hydrofluoric acid, HF (ρ_{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, H_2SO_4 (ρ_{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 [$KHSO_4$] or potassium disulphate [$K_2S_2O_7$].

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):

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

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:

Add the nickel standard solution (4.7 or 4.8) in a 400 ml beaker marked $T_{l,Ni}$ so that the mass fraction of nickel $K_{l,Ni}$ in % is approximately K_{Ni} x 0,92 < $K_{l,Ni}$ < K_{Ni} x 0,98. Select $K_{l,Ni}$ in such a way to take an easy volume with a pipette.

Add the nickel standard solution (4.7 or 4.8) in a 400 ml beaker marked $T_{h,Ni}$ so that the mass fraction of nickel $K_{h,Ni}$ in % is approximately K_{Ni} x 1,02 < $K_{h,Ni}$ < K_{Ni} x 1,08. Select $K_{h,Ni}$ in such a way to take an easy volume with a pipette.

Add to the calibration solutions $T_{l,Ni}$ and $T_{h,Ni}$ all matrix elements whose mass fractions are above 1 % in the test solution, using the appropriate amount of standard solutions (4.9) to match the equivalent matrix composition to the nearest %.

Continue as specified in 7.3: “Add in each flask 15 ml of hydrochloric acid (4.1), 10 ml of nitric acid (4.2)…”.

8 Determination

8.1 Adjustment of the apparatus

Start the inductively coupled plasma optical emission spectrometer and let it stabilize in accordance with the manufacturer’s instructions before taking any measurements.

At one of the wavelengths of the analytical lines listed in Table 1, adjust all appropriate instrumental parameters, as well as the pre-spraying and the integrating times, according to the instrument manufacturer’s instructions while aspirating the highest concentration calibration solution.

Table 1 — Examples of wavelengths for nickel determinations

Wavelength (nm)	Interferences
217,514	/
221,647[a]	Co
222,295[a]	V, Co
222,486	Co
227,021[a]	/
227,877	/
230,299[a]	/
231,234[a]	/
231,604[a]	Co, Mo
239,452	/
341,476[a]	/

[a] These wavelengths were used during the precision test.

Depending on the instrument configuration these parameters may include the outer, intermediate or central gas flow-rates, the torch position, the entrance slits, the exit slits and the photomultiplier tubes voltage.

Other wavelengths may be used, provided that interferences, sensitivity, resolution and linearity criteria have been carefully investigated.

Prepare the software for measurements of the intensity, and for the calculation of the mean value and relative standard deviation corresponding to the appropriate analytical line.

Each time the internal reference element is used, prepare the software to calculate the ratio between the intensity of the analyte and the intensity of the internal reference element.

8.2 Measurement of test solutions

Measure the absolute or ratioed intensity of the analytical line for the lowest calibration solution $T_{l,Ni}$ firstly, then for the test solution T_{Ni} and finally for the highest calibration solution $T_{h,Ni}$. Repeat this sequence three times and calculate the mean intensities $I_{l,Ni}$ and $I_{h,Ni}$ for the low and high calibration solutions and I_{Ni} for the test solution respectively.

9 Expression of the results

9.1 Method of calculation

Calculate the concentration of nickel K_{Ni} in %, in the test solution T_{Ni}, using Formula (2):

$$K_{Ni} = K_{l,Ni} + \frac{(I_{Ni} - I_{l,Ni})(K_{h,Ni} - K_{l,Ni})}{I_{h,Ni} - I_{l,Ni}} \tag{2}$$

9.2 Precision

Twelve laboratories in seven European countries participated in an inter laboratory validation test programme under the auspices of ECISS/TC 102/WG 10, involving three determinations of nickel at nine content levels (samples).

Each laboratory carried out two determinations under repeatability conditions as defined in ISO 5725-1, i.e. one operator, same apparatus, identical operating conditions, same calibration and a minimum period of time. The third determination was carried out on a different day using the same apparatus with a different calibration.

The compositions of the samples used are given in Annex B.

The results obtained were statistically evaluated in accordance with ISO 5725-2, ISO 5725-3 and CEN/TR 10345: they are reported in Table 2.

The logarithmic relationships between the nickel content (m) and the precision parameters (r, R_w and R), together with the corresponding correlation coefficients are:

$\lg r = 0{,}881\,3 \lg m - 1{,}895\,2$ [Correlation coefficient = 0,866]

$\lg R_w = 0{,}937\,0 \lg m - 1{,}836\,7$ [Correlation coefficient = 0,782]

$\lg R = 0{,}660\,1 \lg m - 1{,}241\,5$ [Correlation coefficient = 0,653]

The corresponding graphical representation is shown in Annex C.

Although the correlations above are rather poor, the smoothed values of the repeatability limit (r) and reproducibility limits (R_w and R) of the test results are summarized in Table 3.

10 Test report

The test report shall contain the following information:

a) identification of the test sample;

b) method used (by reference to this European Standard, EN 10361);

c) results;

d) any unusual characteristics noted during the determination;

e) any operation not included in this European Standard or in the document to which reference is made or regarded as optional;

f) date of the test and/or date of preparation or signature of the test report;

g) signature of the responsible person.

Table 2 — Results obtained from the precision test

SAMPLE	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**
Mean (%)	4,833	7,079	8,577	10,119	12,382	14,788	19,758	20,095	24,734
σ (r), %	0,015	0,024	0,037	0,035	0,062	0,041	0,061	0,057	0,073
σ (R_w), %	0,023	0,029	0,038	0,059	0,065	0,047	0,120	0,054	0,123
σ (R), %	0,055	0,060	0,076	0,112	0,164	0,155	0,130	0,096	0,188
r, %	0,043	0,066	0,104	0,099	0,173	0,116	0,171	0,159	0,206
R_w, %	0,065	0,080	0,106	0,164	0,182	0,133	0,336	0,152	0,344
R, %	0,155	0,168	0,214	0,315	0,458	0,434	0,364	0,268	0,520
CV (R)	1,15	0,85	0,89	1,11	1,32	1,05	0,66	0,48	0,75
Aim CV (R)	0,86	0,75	0,70	0,66	0,62	0,58	0,53	0,52	0,49
Max CV (R)	1,88	1,65	1,54	1,46	1,36	1,28	1,15	1,15	1,07

Table 3 — Smoothed values of the repeatability and reproducibility limits

Nickel content % (mass fraction)	**Repeatability limit** *r* (%)	**Reproducibility limits** R_w (%)	 *R* (%)
5	0,052 6	0,065 8	0,165 9
10	0,096 9	0,126 0	0,262 2
20	0,178 4	0,241 2	0,414 3
25	0,217 2	0,297 3	0,480 0

Annex A
(informative)

Plasma optical emission spectrometer - Suggested performance criteria to be checked

A.1 Resolution of a spectrometer

The resolution of a spectrometer can be defined as the wavelength difference, dλ, between two lines which can still just be observed separately. In practice the parameter FWHM (Full Width at Half Maximum) is used as a resolution measure.

Ideally, the resolution should be of the same order as the physical line width in plasma emission spectra, i.e. 2 pm to 5 pm (1 pm = 10^{-12} m). In practice, however, the observed width of the emission lines and, consequently, the resolution will often be determined by the bandwidth of the spectrometer being used. As long as broadening resulting from aberrations can be neglected, this bandwidth is given by:

$$\mathrm{FWHM} = (d\lambda/d\mathrm{x})\ (w_\mathrm{i} + w_\mathrm{u})\ /2 \tag{A.1}$$

where

w_i and w_u are the widths of the entrance slit and exit slit respectively; and

$d\lambda/d\mathrm{x}$ is the reciprocal linear dispersion which is given by:

$d\lambda/d\mathrm{x} = d(\cos\beta)/\mathrm{n}L$.

where

L is the focal length of the spectrometer;

n is the order number;

d is the reciprocal of the groove density in the grating;

β is the diffraction angle.

Normally, commercial spectrometers present resolutions in the range of 4 pm to 30 pm. A good resolution is of great importance to cope with the frequent spectral interferences, which occur in inductively coupled plasma optical emission spectrometry. Since a line with a wavelength in the second order will have the same diffraction angle β as a line with a wavelength 2λ in the first order, a spectrometer should either have an order sorting possibility or an optical filter to avoid an order overlap.

A.2 Short and long-term stability

The evaluation of the short-term stability consists on the calculation of the repeatability standard deviation of a series of measurements carried out with the spectrometer.

For each analyte, a series of ten consecutive intensity measurements of its highest calibration solution is carried out using the typical integration time for the system. The average I_avrg and the standard deviation S_I of the ten measurements are calculated in addition to the relative standard deviation RSD_I, according to the formula:

$$\mathrm{RSD}_\mathrm{I} = (S_\mathrm{I}/I_\mathrm{avrg}) \times 100 \text{ (in \%)} \qquad \text{(A.2)}$$

In inductively coupled plasma optical emission spectrometry, for solutions with concentrations of at least twice the background-equivalent concentration (BEC), RSD_I-values between 0,3 % and 1,0 % are generally accepted. Multi-elemental calibration solutions may be used for measurement at various analytical lines present in simultaneous spectrometers.

Long term stability assessment is a measurement of the instrument drift. This is only required if the spectrometer is set up to work for long intervals of time. It consists of carrying out the same short-term stability tests at specific intervals of time, 15 min to 1 h, and plotting the deviation of the average found for every short-term test against time. Deviations of more than 2 % per hour should not be accepted. In case the instrument is not able to perform better, the control calibration solution should be measured more often during the analysis and the mean results of the test solutions should be recalculated by interpolation between two consecutive control calibration solutions.

Values of short-term stability from 0,36 % to 0,71 % were obtained during the precision test program.

A.3 Evaluating the Background Equivalent

The background equivalent concentration (BEC) is used as an evaluation of the instrument sensitivity. Since the analyte signal has usually a relatively high background, its correction by the background intensity is recommended. It is therefore calculated by using the formula:

$$\mathrm{BEC} = (I_\mathrm{BG} \,/\, I_\mathrm{net}) \text{ x } \mathrm{C}_\mathrm{Analyte} \qquad \text{(A.3)}$$

where

I_BG is the intensity of the background;

I_net is the intensity of the analyte (overall intensity minus intensity of the background);

C_Analyte is the concentration of analyte that yields I_net.

The background equivalent concentration values for the elements to be analysed can be found in wavelength tables (usually part of the instrument software). The background equivalent concentration is the better the smaller its numerical value is.

Values of background equivalent concentration between 0,05 mg/l and 2,3 mg/l were obtained during the precision test program.

Annex B
(informative)

Composition of the samples used for the validation precision test

The compositions of the samples used are listed in Table B.1.

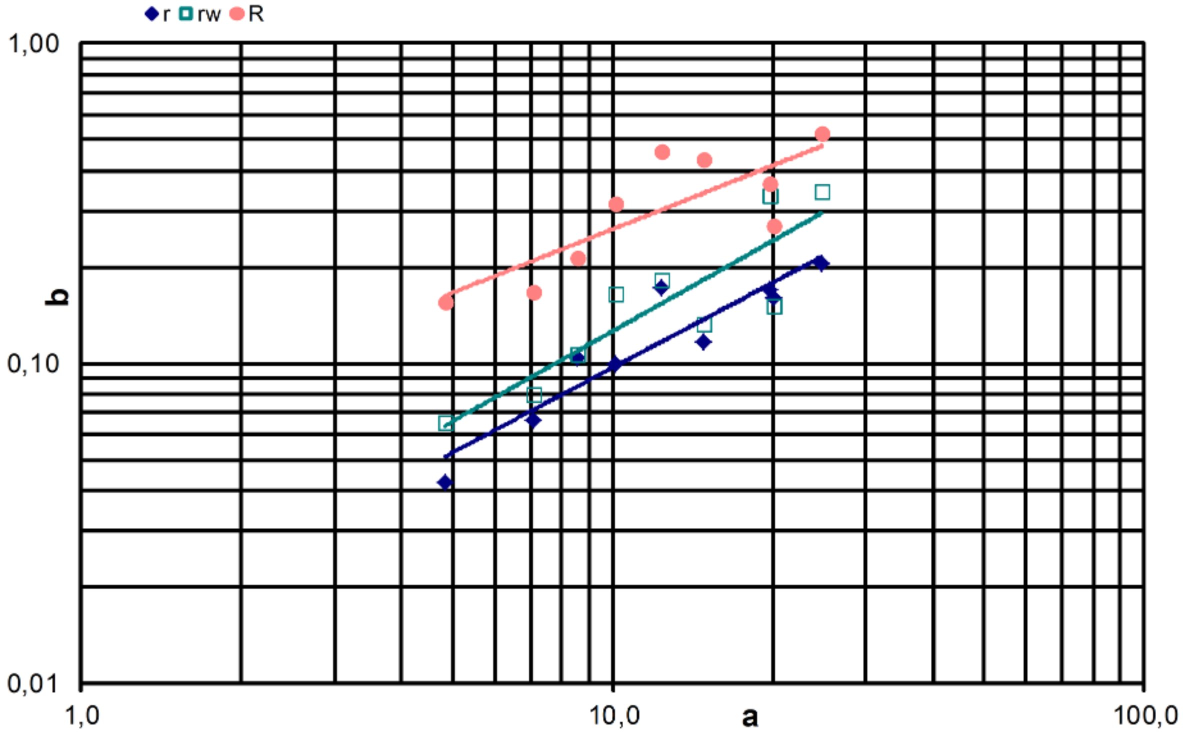
Table B.1 — Compositions of the samples used for the validation precision test

Sample label	Ni	C	Si	Mn	P	S	Cr	Mo	Al	Co	Cu	N	Ti	V	Others
A [ECRM 273–1]	**4,852**	0,0336	0,378	0,785	0,0131	0,0004	14,747	0,246		0,0391	3,047	0,0444		0,0512	As: 0,003, Nb: 0,221, Sn: 0,002
B [ECRM 298–1]	**7,056**	0,0146	0,262	0,398	0,0198	0,0006	24,72	3,799	0,0285	0,0552	0,2008	0,2629	0,0014	0,0607	B: 0,0021, Fe: 63,38
C [ECRM 286–1]	**8,542**	0,1002		1,919	0,0255	0,2801	18,128	0,3285		0,1505		0,0429			Sb: 0,0014 Sn: 0,0084
D [ECRM 231–2]	**10,105**	0,0140	0,3687	1,2625	0,0179	0,0250	18,071	0,3014	0,0032	0,0402	0,0941	0,0444	0,0007	0,0708	As: 0,0048; B: 0,0020, Sb: 0,0011; Sn: 0,0043, W: 0,0141; Ca: 0,0007
E [ECRM 297–1]	**12,335**	0,0223	0,3438	0,8965	0,0135	0,0101	18,369	2,899	0,0195	0,0413	0,2036	0,0152	0,0072	0,0535	As: 0,004 B: 1,4161
F [BCS 474]	**14,74**	0,022	0,17	1,70	0,0080	0,020	19,06	3,55			0,35			0,30	As: 0,030
G [ECRM 327–2]	**19,52**	0,152	2,052	1,289	0,0228	0,0046	24,35	0,174	0,070	0,159	0,060	0,059		0,044	
H [BCS 464/1]	**20,05**	0,086	0,57	0,791	0,020	0,028	25,39			0,54					Pb: 0,0004
I [ECRM 289–1]	**24,68**	0,0489	0,5312	1,0159	0,0114	0,0027	14,63	1,102	0,199	0,0649			2,009	0,2802	B: 0,0044; Sn:0,1108

Annex C
(informative)

Graphical representation of the precision data

Figure C.1 shows the logarithmic relationships between the nickel content (m) and the corresponding repeatability (*r*) and reproducibility parameters (R_w and *R*).



Key

a Ni content % (mass fraction)

b precision (%)

Figure C.1 — Logarithmic relationships between the nickel content (m) and the corresponding repeatability (*r*) and reproducibility parameters (R_w and *R*)

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