



SLOVENSKI STANDARD
oSIST prEN 10178:2023
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Jekla - Določevanje niobija - Spektrofotometrična metoda

Steels - Determination of niobium - Spectrophotometric method

Stähle - Bestimmung von Niob - Photometrisches Verfahren

Aciers - Détermination du niobium - Méthode spectrophotométrique

Ta slovenski standard je istoveten z: prEN 10178

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English Version

Steels - Determination of niobium - Spectrophotometric method

Aciers - Détermination du niobium - Méthode
spectrophotométrique

Stählen - Bestimmung von Niob - Photometrisches
Verfahren

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 459/SC 2.

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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 10178:2023) has been prepared by Technical Committee CEN/TC 459/SC 2 “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 10178:1989.

In comparison with the previous edition, the following technical modifications have been made:

- Normative references: updated;
- Clause 3 Terms and definitions: added;
- Reference 5.13: added;
- Reference 5.18: title simplified;
- Clause 6: developed and sub Clauses 6.1 and 6.2, added;
- Clause 7: updated;
- Former sub Clause 7.3.3: included in 8.3.2;
- Clause 10: updated;
- Bibliography: added.

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prEN 10178:2023 (E)**1 Scope**

This document specifies a spectrophotometric method for the determination of niobium in steels.

The method is applicable to all grades of steels with niobium contents up to 1,3 % (by mass), with a lower limit of detection of 0,002 % (by mass).

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)*

EN ISO 14284, *Steel and iron - Sampling and preparation of samples for the determination of chemical composition (ISO 14284)*

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 <https://www.electropedia.org/>

— ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Principle

Dissolution of a test portion with hydrochloric acid followed by oxidation with hydrogen peroxide. Precipitation of niobium and tantalum with phenylarsonic acid using zirconium as a carrier.

Formation of a complex of niobium with 4-(2-pyridylazo)-resorcinol (PAR) in a buffered sodium tartrate medium.

Spectrophotometric measurement of the coloured compound at a wavelength of 550 nm.

5 Reagents**5.1 General**

During analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696 or water of equivalent purity.

5.2 Iron, of high purity, free from niobium

5.3 Potassium hydrogen sulphate (KHSO₄)

5.4 Hydrochloric acid, ρ_{20} 1,19 g/ml approximately

5.5 Hydrochloric acid, solution 1 + 4

Carefully add 40 ml of hydrochloric acid (5.4) to 160 ml of water and mix.

5.6 Hydrochloric acid, solution 1 + 9

Carefully add 20 ml of hydrochloric acid (5.4) to 180 ml of water and mix.

5.7 Sulphuric acid, solution 1 + 1

Carefully add 100 ml of sulphuric acid (ρ_{20} 1,84 g/ml approximately) to 200 ml of water.

Allow to cool and mix.

5.8 Sulphuric acid, solution 1 + 4

Carefully add 20 ml of sulphuric acid (ρ_{20} 1,84 g/ml approximately) to 80 ml of water.

Allow to cool and mix.

5.9 Ethylenediaminetetraacetic acid di-sodium salt, (EDTA Na₂), 15 g/l solution

Store in polyethylene bottle.

5.10 Hydrogen peroxide, 30 % w/v (100 vol.)**5.11 Phenylarsonic acid**, 40 g/l solution**5.12 Phenylarsonic acid**, 0,5 g/l solution**5.13 4-(2-pyridylazo)-resorcinol mono-sodium salt, (PAR)**, 0,6 g/l solution

The di-sodium salt can also be used but the identical salt shall be used for both calibration and tests.

5.14 Glacial acetic acid, ρ_{20} 1,05 g/ml, approximately**5.15 Sodium acetate buffer**, 350 g/l solution

Dissolve 350 g of sodium acetate trihydrate in 700 ml of water, add 5,5 ml of glacial acetic acid (5.14), dilute to 1 000 ml with water and mix.

Adjust the pH value to 6,3 with small additions of acetic acid (5.14) or sodium hydroxide solution (5.16) using a pH meter (6.1) for the measurement.

5.16 Sodium hydroxide, 120 g/l solution

Store in a polyethylene bottle.

5.17 Tartaric acid, 100 g/l solution**5.18 Zirconium nitrate**, 3 g/l solution in hydrochloric acid medium

Dissolve 0,3 g of zirconium nitrate in 50 ml of hydrochloric acid solution (5.5). Filter through a fine texture filter paper, dilute to 100 ml with water and mix.

5.19 Niobium standard solution, 0,2 g/l

Weigh 0,143 1 g of niobium pentoxide and transfer into a platinum dish. Fuse with 3,5 g of potassium hydrogen sulphate (5.3). Cool and dissolve in 40 ml of tartaric acid (5.17). Add a further 160 ml of tartaric acid (5.17).

Transfer into a 500 ml volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,2 mg of niobium.

6 Apparatus

6.1 Ordinary laboratory equipment

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

6.2 pH meter

6.3 Spectrophotometer, equipped to measure absorbance at a wavelength of 550 nm, with cells of 4 cm or 1 cm optical path length.

7 Sampling

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

8 Procedure

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 on a 1 g test portion of pure iron (5.2), 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 400 ml squat beaker, add 40 ml of hydrochloric acid solution (5.4), cover the beaker with a watch glass and heat until the acid action ceases. Cool slightly and carefully add 5 ml of hydrogen peroxide (5.10). Boil the solution for 1 min, dilute to approximately 200 ml with hot water and add 5 ml of zirconium nitrate solution (5.18).

8.3.2 Separation of the niobium

Heat the solution to boiling and add 25 ml of a boiling solution of phenylarsonic acid (5.11). Boil for 5 min, add a small amount of filter paper pulp, mix well and allow to stand for 10 min.

Filter through a pulp pad prepared from macerated filter paper and remove the adhering particles from the beaker with a rubber-tipped glass rod. Wash the filter alternately with hot hydrochloric acid solution (5.6) and cold phenylarsonic acid solution (5.12) until freed from iron salts. Finally wash several times with cold phenylarsonic acid solution (5.12). Transfer the filter and its content into a silica crucible. Dry, ignite at a temperature as low as possible until all carbonaceous matter is removed, and finally at 800 °C for at least 15 min. Allow to cool in a desiccator, add a few drops of sulphuric acid solution (5.7) and evaporate to dryness very carefully, in order to remove sulphur trioxide.

Add 2 g of potassium hydrogen sulphate (5.3) and fuse carefully until a clear melt is obtained. Allow to cool, dissolve the fusion products with 50 ml of hot tartaric acid (5.17) and transfer the solution into a 400 ml beaker. Add 50 ml of water and mix.

Add 25 ml of sodium hydroxide solution (5.16) and allow to cool. Using a pH meter (6.1) adjust the pH of the solution to approximately 6,0 with either sulphuric acid solution (5.8) or sodium hydroxide solution (5.16) as required. Allow to cool to room temperature, transfer into a 250 ml volumetric flask, dilute to the mark with water and mix.

8.3.3 Development of the colour

Take an aliquot volume of the solution obtained in 8.3.2, according to the instructions given in Table 1.

Table 1 — Volume of the aliquot

Niobium % (by mass)	Volume of the aliquot (ml)
< 0,26	25,0
0,26 to 0,65	10,0
0,65 to 1,30	5,0

Transfer the aliquot into a 100 ml volumetric flask. By means of a pipette add 10 ml of EDTA Na₂ (5.9), 10 ml of PAR (5.13) and 10 ml of buffer solution (5.15), mixing well after each addition. Allow to stand for 15 min at approximately 20 °C, then dilute to the mark with water and mix. Allow to stand for a further 30 min.

8.3.4 Spectrophotometric measurement

Carry out the spectrophotometric measurement at a wavelength of 550 nm after having set the spectrophotometer to zero absorbance with water. Use 4 cm path length cells for niobium contents up to 0,06 % and 1 cm path length cells for contents greater than 0,06 %.

Convert the readings corresponding to the test solution and to the blank test solution to milligrams of niobium by reference to the calibration curve (8.4).

8.4 Establishment of the calibration curve

8.4.1 Preparation of the calibration solutions

Weigh 1,0 g portions of pure iron (5.2) into a series of 400 ml beakers. Add the volumes of the niobium standard solution (5.19) given in Table 2.

Table 2 — Calibration solutions

Niobium standard solution (5.18) ml	Corresponding niobium mass mg	Cell path length cm
0 ^a	0	1 and 4
1,0	0,2	4
2,0	0,4	4
3,0	0,6	1 and 4
5,0	1,0	1
7,0	1,4	1
9,0	1,8	1
11,0	2,2	1
13,0	2,6	1
^a Zero member		

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Continue as described in Clauses 8.3.1 to 8.3.3 but in all cases by taking a 25 ml aliquot in 8.3.3.

8.4.2 Spectrophotometric measurements

Carry out the spectrophotometric measurements according to the first paragraph of subclause 8.3.4, after having adjusted the spectrophotometer to zero absorbance with water.

8.4.3 Plotting the calibration curve

From each of the absorbance readings, subtract the reading corresponding to the zero member.

Prepare the calibration curve by plotting the net absorbance readings against the mass of niobium, expressed in milligrams.

For samples containing more than 0,26 % (by mass) of niobium and for which 10 ml or 5 ml aliquots are used the relationship with the calibration curve based on 25 ml aliquots is given in Table 3.

Table 3 — Relationship with the calibration curve for 10 ml and 5 ml aliquots

Niobium content % (by mass) in 1 g test portion			Cell path length cm
25 ml aliquot	10 ml aliquot	5 ml aliquot	
0	0	0	1 and 4
0,02	-	-	4
0,04	-	-	4
0,06	-	-	1 and 4
0,10	0,25	-	1
0,14	0,35	0,70	1
0,18	0,45	0,90	1
0,22	0,55	1,10	1
0,26	0,65	1,30	1

9 Expression of results

The niobium content, W_{Nb} , expressed as a percentage (%) by mass, is given by Formulae (1) to (2):

$$W_{Nb} = \frac{m_1 - m_0}{m \times 1000} \times \frac{250}{V} \times 100 \quad (1)$$

$$W_{Nb} = \frac{m_1 - m_2}{m} \times \frac{25}{V} \quad (2)$$

where

- m is the mass of the test portion, expressed in grams;
- m_1 is the mass of niobium in the aliquot of the test solution, expressed in milligrams;
- m_0 is the mass of niobium in the blank test solution, expressed in milligrams;
- V is the volume of the aliquot (8.3.3), expressed in ml.