SLOVENSKI STANDARD
SIST EN 10178:1997

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Kemična analiza železovih zlitin - Določevanje niobija v jeklih - Spektrofotometrična metoda

Chemical analysis of ferrous materials - Determination of niobium in steels - Spectrophotometric method

Chemische Analyse von Eisenwerkstoffen - Bestimmung von Niob in Stählen - Photometrisches Verfahren

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Analyse chimique des matériaux sidérurgiques - Dosage du niobium dans les aciers - Méthode spectrophotométrique

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

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Key words : Iron and steel products, chemical analysis, determination of content, niobium, spectrophotometric analysis

English version

Chemical analysis of ferrous materials
Determination of niobium in steels
Spectrophotometric method

Analyse chimique sidérurgiques - Dosage du niobium dans les aciers - Méthode spectrophotométrique

Chemische analyse von Eisenwerkstoffen - Bestimmung von Niob in Stählen - Photometrisches Verfahren

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European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat : Rue Bréderode 2, B-1000 Brussels

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Brief History

This European Standard takes over the content of EURONORM 178-85 "Chemical analysis of ferrous materials - Determination of niobium in steels - Spectrophotometric method", prepared by ECISS/TC 20 "Methods of chemical analysis"; the Secretariat of which is allocated to the Dansk Standardiseringsrad (DS).

It has been submitted to the CEN Formal Vote following the decision of the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization on 1987-11-24/25.

It has been adopted and ratified by CEN BT on 1988-11-05.

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Note in clauses 1 and 9 EURONORM shall read EUROPEAN STANDARD.

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Chemical analysis of ferrous materials
Determination of niobium in steels
Spectrophotometric method

CONTENTS

1. SCOPE AND FIELD OF APPLICATION

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

The method is applicable to all types of steels with niobium contents up to 1.3% (m/m), with a lower detection limit of 0.002% (m/m).

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2. REFERENCE

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EURONORM 18 – Selection and preparation of samples and test pieces for steel and iron and steel products.

3. 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.

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4. REAGENTS

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

4.1 Iron, of high purity, free from niobium

4.2 Potassium hydrogen sulphate ($KHSO_4$)

4.3 Hydrochloric acid, ρ 1.19 g/ml approximately, (12 mol/l approximately)

4.4 Hydrochloric acid, ρ 1.19 g/ml approximately, diluted 1 + 4 (V/V), (2.4 mol/l approximately)

4.5 Hydrochloric acid, ρ 1.19 g/ml approximately, diluted 1 + 9 (V/V), (1.2 mol/l approximately)

4.6 Sulphuric acid, ρ 1.84 g/ml approximately, diluted 1 + 1 (V/V), (9 mol/l approximately)

4.7 Sulphuric acid, ρ 1.84 g/ml approximately, diluted 1 + 4 (V/V), (3.6 mol/l approximately)

4.8 Ethylenediaminetetra-acetic acid di-sodium salt, (EDTA Na_2), 15 g/l solution

Store in a polyethylene bottle.

4.9 Hydrogen peroxide, 30% w/v (100 vol.)

4.10 Phenylarsonic acid, 40 g/l solution

4.11 Phenylarsonic acid, 0.5 g/l solution

4.12 4-(2-pyridylazo)-resorcinol mono-sodium salt, (PAR), 0.6 g/l solution

NOTE – The di-sodium salt may also be used but the identical salt must be used for both calibration and tests.

4.13 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, ρ1.05 g/ml, dilute to 1 000 ml and mix. Adjust the pH value to 6.3 with small additions of acetic acid or sodium hydroxide solution (4.14) using a pH meter for measurement.

4.14 Sodium hydroxide, 120 g/l solution

Store in a polyethylene bottle.

4.15 Tartaric acid, 100 g/l solution

4.16 Zirconium nitrate, 3 g/l solution in hydrochloric acid medium

Dissolve 0.3 g of zirconium nitrate in 50 ml of hydrochloric acid solution (4.4). Filter through a fine filter paper, dilute to 100 ml with water and mix.

4.17 Niobium reference solution, corresponding to 0.2 mg of niobium per millilitre

Weigh 0.1431 g of niobium pentoxide and transfer to a platinum dish. Fuse with 3.5 g of potassium hydrogen sulphate (4.2). Cool and dissolve in 40 ml of tartaric acid solution (4.15). Add a further 160 ml of tartaric acid solution (4.15). Transfer to a 500 ml volumetric flask, dilute to the mark with water and mix.

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5. APPARATUS

Ordinary laboratory equipment and a spectrophotometer suitable for measuring the absorbance of the solution at a wavelength of 550 nm, together with 4 or 1 cm cells.

6. SAMPLING

Sampling shall be carried out in accordance with EURONORM 18.

7. PROCEDURE

7.1 Test portion

Weigh the mass (m) indicated to the nearest 0.001 g:

$$m = 1 \text{ g} \pm 5\%$$

7.2 Blank test

With each analytical run, carry out an analysis on a 1 g portion of pure iron (4.1) in parallel with the test portion

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analysis, using identical reagents, conditions and dilutions throughout.

7.3 Determination

7.3.1 Preparation of the test solution

Transfer the test portion (7.1) to a 400 ml squat beaker, add 40 ml of hydrochloric acid solution (4.3), cover the beaker with a clock glass and heat until solvent action ceases. Cool slightly and add with caution 5 ml of hydrogen peroxide solution (4.9). Boil the solution for 1 min., dilute to approximately 200 ml with warm water and add 5 ml of zirconium nitrate solution (4.16).

7.3.2 Separation of the niobium

Heat the solution to boiling and add 25 ml of a boiling solution of phenylarsonic acid (4.10). 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 adhering particles from the beaker with a rubber-tipped glass rod. Wash the filter alternately with hot dilute hydrochloric acid solution (4.5) and cold phenylarsonic acid solution (4.11) until freed from iron salts. Finally wash several times with cold phenylarsonic acid solution (4.11). Transfer the filter and precipitate to a silica crucible. Dry, ignite at as low a temperature as possible until all carbonaceous matter is removed, and finally at 800 °C for at least 15 min. Cool in a desiccator, add a few drops of sulphuric acid solution (4.6) and evaporate to dryness very carefully. Heat to remove sulphur trioxide.

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7.3.3 Preparation of the solution for spectrophotometry

Add 2 g of potassium hydrogen sulphate (4.2) and fuse carefully until a clear melt is obtained. Cool and dissolve the fusion products with 50 ml of a warm solution of tartaric acid (4.15) and transfer to a 400 ml beaker. Add 50 ml of water and mix.

Add 25 ml of sodium hydroxide solution (4.14) and cool. By means of a pH meter adjust the pH of the solution to approximately 6.0 with either sulphuric acid solution (4.7) or sodium hydroxide solution (4.14) as required. Cool to room temperature, transfer to a 250 ml volumetric flask, dilute to the mark with water and mix.

7.3.4 Development of the colour

Take an aliquot volume of the test solution (7.3.3) according to the following table:

Niobium % (m/m)	*Volume of aliquot (ml)*
Less than 0.26	25.0
0.26-0.65	10.0
0.65-1.30	5.0

Transfer the aliquot to a 100 ml volumetric flask. By means of a pipette add 10 ml of EDTA Na_2 solution (4.8), 10 ml of PAR solution (4.12) and 10 ml of buffer solution (4.13), 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.

7.3.5 Spectrophotometric measurement

Carry out the spectrophotometric measurement at a wavelength of 550 nm after having set the spectrophotometer to zero absorbance in relation to water. Use 4 cm cells for niobium contents up to 0.06% and 1 cm cells for contents greater than 0.06%.

Convert the readings for the test portion solution and for the blank test solution to milligrams of niobium by reference to the calibration graph (7.4).

7.4 Establishment of the calibration graph

7.4.1 Preparation of the calibration solutions

Weigh 1.0 g portions of pure iron (4.1) into a series of 400 ml beakers. Add from a burette, volumes of niobium reference solution (4.17) as shown in the table below:

Niobium solution ml	Niobium added mg	Cell size
0	0	1 and 4 cm
1.0	0.2	4 cm
2.0	0.4	4 cm
3.0	0.6	1 and 4 cm
5.0	1.0	1 cm
7.0	1.4	1 cm
9.0	1.8	1 cm
11.0	2.2	1 cm
13.0	2.6	1 cm

Continue as described in clauses 7.3.1 to 7.3.4 but in all cases taking a 25 ml aliquot in 7.3.4.

7.4.2 Spectrophotometric measurements

Carry out spectrophotometric measurements according to the method described in paragraph 1 of clause 7.3.5, after having adjusted the spectrophotometer to zero absorbance in relation to water.

7.4.3 Plotting the calibration graph

From each of the absorbance readings, subtract the reading obtained on the test portion with no added niobium. Prepare a calibration graph by plotting the net absorbance readings against milligrams of niobium.

NOTE: For test samples containing more than 0.26% 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 as follows:

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Equivalent % Nb on 1 g sample			Cell size
25 ml aliquot	10 ml aliquot	5 ml aliquot	
0	0	0	1 and 4 cm
0.02	–	–	4 cm
0.04	–	–	4 cm
0.06	–	–	1 and 4 cm
0.10	0.25	–	1 cm
0.14	0.35	0.70	1 cm
0.18	0.45	0.90	1 cm
0.22	0.55	1.10	1 cm
0.26	0.65	1.30	1 cm

8. EXPRESSION OF RESULTS

The percentage by mass of niobium (Nb) is given by the expression

$$\text{Nb\% (m/m)} = \frac{m_1 - m_0}{m \cdot 1000} \cdot \frac{250}{V} \cdot 100 = \frac{m_1 - m_0}{m} \cdot \frac{25}{V}$$

where:

m is the mass, in grams, of the test portion;

m_1 is the mass, in milligrams, of niobium found in the aliquot of the test portion solution;

m_0 is the mass in milligrams of niobium found in the blank test solution;

V is the aliquot volume, in millilitres, taken from the test portion solution.

9. TEST REPORT

The test report shall contain the following particulars:

(a) the method of analysis used by reference to this EURONORM;

(b) the results obtained, as well as the form in which they are expressed;

(c) any particular details which may have been noted during the determination;

(d) any operations not specified in this EURONORM or any optional operations which could have had an influence on the result;

(e) all indications necessary for the identification of the sample;

(f) the laboratory and the date of analysis.

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ANNEX

Precision data

Planned trials of this method were carried out by 10 analysts from different laboratories; 5 determinations were carried out by each analyst on each 4 samples; 5 determinations were made by 7 of the analysts on a fifth sample.* From the results obtained the 95% (2s) confidence limits have been calculated in accordance with ISO 5725, and are tabulated as follows:

Alloy type	Niobium % (m/m)	Repeatability r	Reproducibility R
Non-alloy steel	0.029	0.0020	0.0024
Non-alloy steel	0.099	0.0051	0.0076
Magnet alloy, 13% Ni, 8% Al, 3% Cu, 25% Co	0.589	0.0127	0.0212
Niobium stabilized steel 18% Cr, 9.5% Ni	1.035	0.0229	0.0408
Niobium stabilized steel 17.5% Cr, 13% Ni *	0.906	0.0175	0.0416

Repeatability

The difference between two single results found on identical material by one analyst using the same apparatus within a short time interval will exceed the repeatability, r, not more than once in 20 cases in the normal and correct operation of the method.

Reproducibility

The difference between two single and independent results found by two operators working in different laboratories on identical test material will exceed the reproducibility, R, on average, not more than once in 20 cases in the normal and correct operation of the method.