

# **SLOVENSKI STANDARD**

## **SIST EN 10211:2014**

**01-julij-2014**

**Nadomešča:**

**SIST EN 10211:1997**

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### **Kemična analiza železovih zlitin - Določevanje titana v jeklih in železovih litinah - Plamenska atomska absorpcijska spektrometrična metoda**

Chemical analysis of ferrous materials - Determination of titanium in steels and cast irons  
- Flame atomic absorption spectrometric method

Chemische Analyse von Eisenwerkstoffen - Bestimmung des Titananteils in Stahl und  
Eisen - Flammenatomabsorptionsspektrometrisches Verfahren

Analyse chimique des produits ferreux - Détermination du titane dans les aciers et les  
fontes - Méthode par spectrométrie d'absorption atomique dans la flamme

**Ta slovenski standard je istoveten z: EN 10211:2013**

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#### **ICS:**

77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.080.01	Železne kovine na splošno	Ferrous metals in general

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

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**Chemical analysis of ferrous materials - Determination of  
titanium in steels and cast irons - Flame atomic absorption  
spectrometric method**

Analyse chimique des produits ferreux - Détermination du  
titane dans les aciers et les fontes - Méthode par  
spectrométrie d'absorption atomique dans la flamme

Chemische Analyse von Eisenwerkstoffen - Bestimmung  
des Titananteils in Stahl und Eisen -  
Flammenatomabsorptionsspektrometrisches Verfahren

This European Standard was approved by CEN on 29 September 2013.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
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## Foreword

This document (EN 10211:2013) has been prepared by Technical Committee ECISS/TC 102 “Methods of chemical analysis for 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of an identical text or by endorsement, at the latest by May 2014, and conflicting national standards shall be withdrawn at the latest by May 2014.

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.

This document supersedes EN 10211:1995.

In comparison with the previous edition of EN 10211, the following technical changes were made:

- Clause 1, “steel and iron” replaced by “non-alloyed and alloyed steels and cast irons”;
- Clause 3, clarification regarding the use of aluminium chloride within both dissolutions described;
- Addition of 5.2.3 (information on the linearity criteria);
- Table 1, addition of terms corresponding to 0,025 % and 0,075 % titanium;
- Subclause 7.6 was merged in Subclause 7.5;
- Clause 8, formula: notation changed;
- Annex A, the notes were included in the text and the correlation coefficients corrected;
- Annex B: added.

According to the CEN-CENELEC Internal Regulations, the national standards organisations 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.

## EN 10211:2013 (E)

## 1 Scope

This European Standard specifies a flame atomic absorption spectrometric method for the determination of titanium in steels and cast irons. The method is applicable to non-alloyed and alloyed steels and cast irons with titanium contents of 0,01 % to 1,0 % (m/m).

## 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)*

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

## 3 Principle

Two dissolution procedures are possible and described:

- a) The first one is specific to cast irons and is based on dissolution of the test portion with nitric and perchloric acids, followed by evaporation with perchloric acid to dryness. The further steps being a separation of the insolubilised silica by filtration and collection of the filtrate.
- b) The second one, which can be used for steels as well as for cast irons is based on dissolution of the test portion with hydrochloric and nitric acid. This dissolution is followed by:
  - 1) filtration and ignition of the acid insoluble residue and removal of silica with hydrofluoric acid;
  - 2) fusion of the residue with a mixture of boric acid and sodium carbonate, dissolution of the melt with acid and addition of this solution to the reserved filtrate.

Addition of aluminium chloride as interaction buffer, in both cases.

Nebulisation of the test solution into a slightly fuel rich nitrous oxide/acetylene flame, of an atomic absorption spectrometer.

Spectrometric measurement of the atomic absorption of the 365,3 nm (or 364,3 nm) spectral line emitted by a titanium hollow-cathode lamp.

## 4 Reagents

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

**4.1 Nitric acid**,  $\text{HNO}_3$  ( $\rho_{20} = 1,40$  g/ml).

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

**4.3 Perchloric acid**,  $\text{HClO}_4$  ( $\rho_{20} = 1,67$  g/ml).

**4.4 Sulphuric acid**,  $\text{H}_2\text{SO}_4$  ( $\rho_{20} = 1,84$  g/ml).

**4.5 Hydrofluoric acid**,  $\text{HF}$  ( $\rho_{20} = 1,15$  g/ml).

**4.6 Nitric acid solution**, 1 + 1.

Add 100 ml of nitric acid (4.1) to 100 ml water

**4.7 Hydrochloric acid solution**, 1 + 1.

Add 100 ml of hydrochloric acid (4.2) to 100 ml water

**4.8 Aluminium chloride solution**, equivalent to 10 g/l Al.

Dissolve 90 g of hydrated aluminium chloride ( $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ ) in a 600 ml beaker with water and transfer the solution into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

**4.9 Fusion mixture.**

Mix one part by mass of boric acid,  $\text{H}_3\text{BO}_3$  and one part of sodium carbonate anhydrous,  $\text{Na}_2\text{CO}_3$ .

Store in a stoppered polyethylene container.

**4.10 Iron-perchloric matrix solution**, 20 g/l Fe.

Weigh, to the nearest 0,01 g, 10,00 g of pure iron free of titanium or of known low titanium content. Transfer into a 600 ml beaker and cover with a watch glass. Add in small portions, 150 ml of nitric acid (4.6). When effervescence ceases, add 200 ml of perchloric acid (4.3). Heat gently, until the iron is completely dissolved.

Continue heating at a temperature at which a steady reflux of white perchloric acid fumes is maintained on the walls of the beaker. Continue heating until there are no perchloric acid fumes visible inside the beaker and the iron perchlorate crystallises.

Allow to cool, add 150 ml of water, swirl and boil gently to dissolve the salts.

Transfer quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

**4.11 Iron-chloro nitric matrix solution**, 20 g/l Fe.

Weigh, to the nearest 0,01 g, 20,00 g of pure iron free of titanium or of known low titanium content. Transfer into a 600 ml beaker, add 200 ml of water and then 220 ml of hydrochloric acid (4.2), in small portions. Cover with a watch glass and heat gently until effervescence ceases. Add gradually 60 ml of nitric acid (4.1). Heat for about 10 min at about 80 °C, to expel nitrous fumes.

Allow to cool and transfer quantitatively into a 1 000 ml one-mark volumetric flask. Add water until about 600 ml and add 20 g of fusion mixture (4.9). Dissolve, dilute to the mark with water and mix.

**4.12 Titanium, 1 g/l standard solution.**

Weigh ( $1 \pm 0,001$ ) g of titanium (99,99 % purity), transfer into a 400 ml beaker and cover with a watch glass. Dissolve it in 200 ml of hydrochloric acid solution (4.7) and add two drops of hydrofluoric acid (4.5).

Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

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1 ml of this solution contains 1 mg of titanium.

**4.13 Titanium, 0,5 g/l standard solution [freshly prepared].**

Transfer 50,0 ml of titanium standard solution (4.12) into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this standard solution contains 0,5 mg of titanium.

**4.14 Titanium, 0,05 g/l standard solution [freshly prepared].**

Transfer 10,0 ml of titanium standard solution (4.12) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this standard solution contains 0,05 mg of titanium.

**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 30 ml platinum crucibles.****5.2 Atomic absorption spectrometer.**

This shall be equipped with a titanium hollow-cathode lamp and supplied with nitrous oxide and acetylene sufficiently pure to give a clear, steady slightly fuel-rich flame, free from water and oil, and free from titanium.

The atomic absorption spectrometer used will be satisfactory if, after optimisation according to 7.5, the limit of detection and characteristic concentration are in reasonable agreement with the values given by the manufacturer and it meets the precision criteria given in 5.2.1 to 5.2.3.

It is also recommended that the instrument should conform to the additional performance requirements given in 5.2.4.

**5.2.1 Minimum precision.**

Calculate the standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution. The standard deviation shall not exceed 1,5 % of the mean absorbance of this solution.

Calculate the standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution (excluding the zero member). The standard deviation shall not exceed 0,5 % of the mean absorbance of the most concentrated calibration solution.

**5.2.2 Limit of detection.**

The limit of detection is a number, expressed in units of concentration (or amount) that describes the lowest concentration level (or amount) of an element that can be determined to be statistically different from an analytical blank.

The limit of detection of titanium in a matrix similar to that of the final test portion solution shall be better than 0,2 µg/ml for the 365,3 nm line and better than 0,1 µg/ml for the 364,3 nm line.



### 5.2.3 Linearity of calibration.

The value of the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) shall not be less than 0,7 times the value of the slope for the bottom 20 % of the concentration range (expressed as a change in absorbance) determined in the same way.

For instruments with automatic calibration using two or more standard solutions, it shall be established prior to the analysis, by obtaining absorbance readings, that the above requirements for graph linearity are fulfilled.

### 5.2.4 Characteristic concentration.

The characteristic concentration for titanium in a matrix similar to the final test portion solution shall be better than 1,9 µg/ml for the 365,3 nm line and better than 1,4 µg/ml for the 364,3 nm line.

## 5.3 Ancillary equipment.

A system for acquisition of the measured values, data processing and evaluation is necessary. Usually data processing and evaluation is performed with the appropriate software of the computer, connected to the atomic absorption spectrometer.

Scale expansion can be used until the noise observed is greater than the read-out error and is preferred for absorbances below 0,1. If scale expansion has to be used and the instrument does not have the means to read the value of the scale expansion factor, the value can be calculated by measuring the absorbances of a suitable solution with and without scale expansion and using the ratio of the signals obtained.

## 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 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all reagents as used for the determination in accordance with the chosen dissolution method, 7.3.1 or 7.3.2, including iron (solution 4.10 or 4.11 depending on the chosen dissolution).

### 7.3 Preparation of the test solution

#### 7.3.1 Nitric perchloric dissolution

Transfer the test portion (7.1) into a 250 ml beaker. Cover the beaker with a watch glass and add 15 ml of nitric acid (4.6). When effervescence ceases, add 20 ml of perchloric acid (4.3). Heat gently until the reaction dissolution is achieved.

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Continue heating at a temperature at which a steady reflux of white perchloric acid fumes is maintained on the walls of the beaker. Continue heating until there are no perchloric acid fumes visible inside the beaker and the iron perchlorate crystallises.

Allow to cool, add 25 to 30 ml of water, swirl and boil gently for a few minutes. Filter the silica through a medium-texture filter paper and collect the filtrate in a 100 ml volumetric flask. Wash the filter with hot water several times and collect the washings in the same 100 ml volumetric flask.

Allow to cool and add 5 ml of aluminium chloride solution (4.8). Dilute to the mark with water and mix.

**7.3.2 Chloro-nitric dissolution with treatment of the residue**

Transfer the test portion (7.1) into a 250 ml beaker. Add 20 ml of hydrochloric acid (4.7), cover the beaker with a watch glass and heat gently until effervescence ceases. Add gradually 3 ml of nitric acid (4.1). Heat for about 10 min, at about 80 °C to expel nitrous fumes. Allow to cool.

Filter through a dry filter and collect the filtrate in a 100 ml volumetric flask. Wash the residue on the filter with boiling water (about 40 ml).

Transfer the filter into a platinum crucible (5.1) and cover with a suitable platinum cover.

Place the covered crucible progressively into a muffle furnace set at 900 °C in order to ash the filter and the residue. Allow to stand at 900 °C for about 15 minutes, then remove and allow to cool. Add into the crucible 2 drops of sulphuric acid (4.4) and 2 ml of hydrofluoric acid (4.5). Evaporate to dryness on a hotplate and cool.

Add 1,0 g of the fusion mixture (4.9). Fuse the content of the crucible in a muffle furnace at 1 000 °C for 15 min to 20 min. Allow the crucible to cool and add about 10 ml of water and 1 ml of hydrochloric acid (4.2) to the solidified melt. Heat gently, in order to dissolve the fusion products. Allow the crucible to cool and add this solution quantitatively to the filtrate in the 100 ml volumetric flask. Dilute to the mark with water and mix.

**7.4 Preparation of the calibration solutions**

Transfer 50 ml of iron solution (4.10) or (4.11) according to the chosen method of dissolution (7.3.1 or 7.3.2) into each of a series of 100 ml volumetric flasks and add 5 ml of aluminium chloride solution (4.8). Using a burette or a pipette, add the volumes of titanium standard solutions (4.13 or 4.14) shown in Table 1. Dilute to the mark with water and mix.

The proposed calibration is given as a guide. It should be adapted in accordance with the sensitivity of the instrument used.

Table 1 — Calibration solutions

Titanium standard solution volume ml	Corresponding titanium mass mg	Iron mass mg	Corresponding content of titanium in the test sample % (m/m)
0 <sup>a</sup>	0	1 000	0
2,0 (4.14)	0,10	1 000	0,010
5,0 (4.14)	0,25	1 000	0,025
10,0 (4.14)	0,50	1 000	0,050
15,0 (4.14)	0,75	1 000	0,075
20,0 (4.14)	1,00	1 000	0,100
40,0 (4.14)	2,00	1 000	0,200
6,0 (4.13)	3,00	1 000	0,300
10,0 (4.13)	5,00	1 000	0,500
14,0 (4.13)	7,00	1 000	0,700
20,0 (4.13)	10,00	1 000	1,000
<sup>a</sup> Zero member			

### 7.5 Adjustment of atomic absorption spectrometer

Fit the titanium hollow-cathode lamp into the atomic absorption spectrometer (5.2), switch on the current and allow it to stabilise. Adjust the wavelength in the region of 365,3 nm or 364,3 nm to minimum absorbance. Following the manufacturer's instructions, fit the correct burner, light the flame and allow the burner temperature to stabilise.

Adjust the flame to be slightly fuel-rich and the burner height to about 1 cm below the light path.

Aspirate the calibration solution containing the highest content of the analyte and adjust the burner configuration and gas flows to obtain the maximum absorption. It is important to ensure that the flow rate of the acetylene is greater than the minimum flow rate advised by the instrument manufacturer.

Evaluate the criteria of 5.2.1 to 5.2.4 to ensure that the instrument and the chosen wavelength are suitable for the determination by measuring the absorbance of the different solutions required by reference to water.

**WARNING** —The manufacturer's recommendations should be closely followed and particular attention is drawn to the following safety points, in particular for solutions containing perchloric acid:

- Aspirate all solutions for the shortest practical period;
- Always aspirate distilled water between each solution. Minimise the aspiration of air;
- Clean the burner frequently. Never allow the burner to clog and wash it thoroughly both inside and out;
- Ensure that all safety covers are in position and wear approved ear protectors and safety glasses.

### 7.6 Spectrometric measurements of the calibration solutions

Aspirate the series of calibration solutions (7.4) in succession, into the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration curve. Spray