

BS EN 10211:2013



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

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee ISE/102, Methods of Chemical Analysis for Iron and Steel.

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

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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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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of an identical text or by endorsement, at the latest by May 2014, and conflicting national standards shall be withdrawn at the latest by May 2014.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.

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, HNO_3 ($\rho_{20} = 1,40 \text{ g/ml}$).

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

4.3 Perchloric acid, HClO_4 ($\rho_{20} = 1,67 \text{ g/ml}$).

4.4 Sulphuric acid, H_2SO_4 ($\rho_{20} = 1,84 \text{ g/ml}$).

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

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, H_3BO_3 and one part of sodium carbonate anhydrous, Na_2CO_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.