SLOVENSKI STANDARD
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 steel and iron - Flame atomic absorption spectrometric method

Chemische Analyse der Eisen- und Stahlwerkstoffe - Bestimmung des Titaniumgehaltes in Stahl und Eisen - Flammenatomabsorptionspektrometrisches Verfahren

Analyse chimique des matériaux sidérurgiques - Dosage du titane dans les aciers et les fontes - Méthode par spectrométrie d'absorption atomique

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English version

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CEN

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

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Foreword

This European Standard has been prepared by of the Technical Committe ECISS/TC 20 "Methods of chemical analysis" of which the secretariat 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of an identical text or by endorsement, at the latest by April 1996, and conflicting national standards shall be withdrawn at the latest by April 1996.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

1 Scope

This European Standard specifies a flame atomic absorption spectrometric method for the determination of titanium in steel and iron.

The method is applicable to alloyed and non-alloyed steel and iron with titanium contents of 0,01% to 1% (m/m).

2 Normative References

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publicatation referred to applies.

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

INFORMATION CIRCULAR No. 8: Recommendations for the drafting of standard methods of analysis employing flame atomic absorption spectrometry for the chemical analysis of iron and steel.

INFORMATION CIRCULAR No. 9: Operational guidelines for the application of flame atomic absorption spectrometry in standard methods for the chemical analysis of iron and steel.

3 Principle

Two dissolution procedures are possible:

3.1 Dissolution with nitric and perchloric acid

Separation of the silica by filtration, after rendering insoluble by evaporation to dryness in perchloric medium.

3.2 Dissolution with hydrochloric and nitric acid and recovery of the residue by fusion with a mixture of sodium carbonate and boric acid

Addition of aluminium chloride as interaction buffer.

Nebulisation of the solution in a nitrous oxide-acetylene flame, slightly fuel rich, and comparison of the absorbance of the resonance energy of the titanium present in the test solution with

that in the calibration solutions.

Recommended wavelength: 365,3 nm (or 364,3 nm)

4 Reagents

During the analysis, use only reagents of recognised analytical reagent quality and having a very low titanium content, and only distilled water or water of equivalent purity.

4.1 Nitric acid, approximately 1,40 g/ml.

4.2 Hydrochloric acid, approximately 1,19 g/ml.

4.3 Perchloric acid, approximately 1,67 g/ml.

4.4 Sulphuric acid, approximately 1,84 g/ml.

4.5 Hydrofluoric acid, approximately 1,15 g/ml.

4.6 Nitric acid, approximately 1,40 g/ml, diluted 1 + 1.

4.7 Hydrochloric acid, approximately 1,19 g/ml, diluted 1 + 1.

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

Dissolve 90 g of hydrated aluminium chloride ($AlCl_3.6H_2O$) in water. Transfer to a 1000 ml flask. Make up to volume with water and mix.

4.9 Flux mixture

Mix intimately one part of sodium carbonate (Na_2CO_3) with an equal part by mass of boric acid (H_3BO_3).

Store in a stoppered polyethylene container.

4.10 Iron, 20 g/l perchloric solution

Weigh to the nearest 0,01 g, 10 g of pure iron free of titanium or of known low titanium content. Place in a 600 ml beaker and cover with a watch glass. Add, in small amounts, 150 ml of nitric acid (4.6). After effervescence ceases, add 200 ml of perchloric acid (4.3). Heat gently to complete the attack. When this is finished, raise the temperature to white perchloric fumes and continue heating until crystallisation of iron perchlorate. Allow to cool.

Take up in about 150 ml of water. Swirl to dissolve the salts, bring gently to the boil. Cool. Transfer quantitatively to a 500 ml volumetric flask. Make up to volume with water and mix.

4.11 Iron, 20 g/l chloro-nitric solution

Weigh to the nearest 0,01 g, 20 g of pure iron free of titanium or of known titanium content. Place in a 600 ml tall form beaker, then add 200 ml of water and gradually 220 ml of hydrochloric acid (4.2). Cover with a watch glass and heat carefully until dissolution is complete. After effervescence ceases, add gradually 60 ml of nitric acid (4.1). Heat for 10 minutes at about 80°C to eliminate nitrous fumes. Remove from the heat and allow to cool. Transfer to a 1000 ml volumetric flask. Dilute to about 600 ml. Add 20 g of flux mixture (4.9). Dissolve, make up to volume with water and mix.

4.12 Titanium, 1 g/l standard solution

Weigh to the nearest 0,001 g, 1 g of pure titanium (99,99%). Place in a 400 ml beaker, cover with a watch glass. Dissolve in 200 ml hydrochloric acid (4.7); add two drops of hydrofluoric acid (4.5). Heat to complete the dissolution. Cool. Transfer the solution quantitatively to a 1000 ml volumetric flask. Make up to volume with water and mix.

1 ml of this standard solution contains 1 mg of titanium.

4.13 Titanium, 0,05 g/l standard solution (to be prepared immediately before use)

Take exactly 10 ml of the titanium standard solution (4.12). Place in a 200 ml volumetric flask. Make up to volume with water and mix.

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

4.14 Titanium, 0,5 g/l standard solution (to be prepared immediately before use)

Take exactly 50 ml of the titanium standard solution (4.12). Place in a 100 ml volumetric flask. Make up to volume with water and mix.

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

5 Apparatus

Ordinary laboratory equipment and

5.1 30 ml platinum crucible

5.2 Atomic absorption spectrometer, a titanium hollow cathode lamp; supplies of nitrous oxide and acetylene sufficiently pure to give a clear, steady slightly fuel-rich flame, free of water and oil and free of titanium.

The atomic absorption spectrometer used will be satisfactory if, after optimization according to 7.6, the limit of detection and characteristic concentration are in reasonable agreement with the values given by the manufacturer and it meets the following performance requirements.

5.2.1 Minimum precision

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

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

5.2.2 Additional performance requirements

It is also desirable that the instrument should conform to the following additional performance requirements (See clause 2 of Information Circular No. 8).

a) 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.

b) Limit of detection - 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.

5.3 Ancillary equipment

A strip chart recorder and/or digital readout device is recommended to evaluate the criteria in 5.2 and for all subsequent measurements.

6 Sampling

Sampling shall be carried out in accordance with EURONORM 18.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,001 g, a test portion of 1 g ± 0,01 g.