SLOVENSKI STANDARD
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Kemiļna analiza ģelezovih zlitin ï Doloļevanje fosforja v malolegiranih jeklih in ģelezovih litinah ï Spektrofotometriļna metoda z molibdenovim modrim

Chemical analysis of ferrous materials - Determination of phosphorus in non-alloyed steels and irons - Molybdenum blue spectrophotometric method

Chemische Analyse von Eisenwerkstoffen - Bestimmung von Phosphor in unlegierten Stählen und Eisen - Spektralphotometrisches Verfahren über Molybdänblau

Analyse chimique des matériaux sidérurgiques - Dosage du phosphore dans les aciers et fontes non alliés - Méthode par spectrophotométrie d´absorption moléculaire

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Chemical analysis of ferrous materials - Determination of phosphorus in non-alloyed steels and irons - Molybdenum blue spectrophotometric method

Analyse chimique des matériaux sidérurgiques - Détermination du phosphore dans les aciers et fontes non alliés - Méthode par spectrophotométrie au bleu de molybdène

Chemische Analyse von Eisenwerkstoffen - Bestimmung von Phosphor in unlegierten Stählen und Eisen - Spektralphotometrisches Verfahren über Molybdänblau

This European Standard was approved by CEN on 30 December 2005.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This European Standard (EN 10184:2006) has been prepared by Technical Committee ECISS/TC 20 “Methods of chemical analysis of ferrous products”, 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of an identical text or by endorsement, at the latest by August 2006, and conflicting national standards shall be withdrawn at the latest by August 2006.

This European Standard supersedes EN 10184:1989.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard specifies a method for the molybdenum blue spectrophotometric determination of phosphorus in non-alloyed steels and irons.

The method is applicable to non-alloyed steels and irons with phosphorus contents from 0,005 % to 0,25 % (m/m).

2 Normative references

The following referenced documents are indispensable for the application 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 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition (ISO 14284:1996)*

3 Principle

Dissolution of a test portion in nitric and hydrochloric acids and controlled addition of perchloric acid.

Formation of the phosphomolybdate complex after removal of silicon and arsenic and reduction with hydrazine sulphate to molybdenum blue.

Spectrophotometric measurement of the blue complex at a wavelength of 680 nm or 825 nm.

4 Reagents

4.1 General

During the analysis, unless otherwise stated, only reagents of recognized analytical grade shall be used and only distilled water or water of equivalent purity.

Blank tests shall verify that the relevant reagents are free from phosphorus. Whenever necessary, the results shall be corrected accordingly. Grades giving high blank values are unsuitable and should be discarded.

4.2 Hydrochloric acid

ρ about 1,19 g/ml.

4.3 Nitric acid

ρ about 1,40 g/ml.

4.4 Perchloric acid

ρ about 1,67 g/ml.

4.5 Hydrofluoric acid

ρ about 1,13 g/ml, diluted 1 + 9.

4.6 Hydrobromic acid

ρ about 1,47 g/ml, diluted 1 + 1.

4.7 Perchloric acid

ρ about 1,67 g/ml, diluted 42 + 58.

420 ml perchloric acid (4.4) shall be carefully poured into 400 ml of water. Swirl, cool, transfer to a 1000 ml volumetric flask. Dilute to the mark with water and mix.

4.8 Sulphuric acid

ρ about 1,84 g/ml, diluted 3 + 37.

37,5 ml sulphuric acid (ρ about 1,84 g/ml) shall be carefully poured into 300 ml water. Swirl, cool and transfer to a 500 ml volumetric flask. Dilute to the mark with water and mix.

4.9 Sodium metabisulphite

100 g/l solution.

100 g of dry sodium metabisulphite ($Na_2S_2O_5$) shall be dissolved in 500 ml of warm water, filter and collect the filtrate in a 1000 ml volumetric flask. Cool, dilute to the mark with water and mix.

4.10 Hydrazine sulphate

1,5 g/l solution to be prepared immediately before use.

4.11 Ammonium molybdate

20 g/l solution.

Dissolve 20 g of ammonium molybdate [$(NH_4)_6Mo_7O_{24}4H_2O$] in about 100 ml of diluted sulphuric acid (4.8). Dilute to around 500 ml with water. Carefully pour 300 ml of sulphuric acid (4.8) into this solution. Swirl, cool and transfer to a 1000 ml volumetric flask. Dilute to the mark with water and mix.

4.12 Molybdate reagent

Prepare immediately before use the following mixture:

Add, in order, to a 1000 ml volumetric flask, swirling between additions:

— 500 ml of water;

— 250 ml of molybdate solution (4.11);

— 100 ml of hydrazine sulphate solution (4.10).

Dilute to the mark with water and mix.

4.13 Phosphorus

5 mg/l standard solution.

Weigh, to the nearest 0,0001 g, 0,4393 g of potassium dihydrogen phosphate (KH_2PO_4), previously dried to constant mass at 105 °C and cooled in a desiccator. Dissolve in water and add 40 ml perchloric acid, ρ about 1,67 g/ml, diluted 1 + 5. Transfer to 1000 ml volumetric flask, dilute to the mark with water and mix.

Transfer 50,0 ml of this solution to a 1000 ml volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 5 μg of phosphorus.

5 Apparatus

Ordinary laboratory equipment and spectrophotometer, suitable for measuring the absorbance of the solution at a wavelength of 680 nm or 825 nm, together with 2 cm or 1 cm optical cells.

6 Sampling

Sampling shall be carried out in accordance with EN ISO 14284.

Chips of thickness less than 2 mm.

7 Procedure

7.1 Test portion

Weigh a mass (m) to the nearest 0,001 g:

m = 1 g ± 5 % for 0,005 < P (%) < 0,050,

m = 0,5 g ± 5 % for 0,050 < P (%) < 0,1,

m = 0,2 g ± 5 % for 0,1 < P (%) < 0,25.

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 the reagents.

7.3 Determination

7.3.1 Preparation of test solution

Introduce the test portion (7.1) into a wide necked 250 ml conical flask. Add 10 ml of nitric acid (4.3), 20 ml of hydrochloric acid (4.2) and 5 ml of water. Cover with a watch-glass and warm gently until dissolution is complete. Remove the watch-glass and add 17 ml of perchloric acid (4.4). Heat until the appearance of perchloric acid fumes. Remove from the heat and allow to cool.

Add 10 ml of hydrofluoric acid (4.5). Heat again until the appearance of perchloric acid fumes. Remove from the heat. Allow to cool (see the note in Clause 9).

Add 10 ml of hydrobromic acid (4.6). Heat again to perchloric acid fumes. When the fumes collect at the bottom of the cylindrical part of the neck of the 250 ml conical flask, maintain for about 1 min.

Withdraw from the heat source.

Add 30 ml of water and heat to dissolve the salts. Bring to the boil, then remove from the source of heat and allow to cool. Transfer to a 100 ml volumetric flask, dilute to the mark with water and mix.

7.3.2 Formation of the reduced phosphomolybdate complex

Transfer a 20 ml aliquot of the solution, obtained in 7.3.1, into each of two wide-necked, 250 ml conical flasks, the one to be used as the test solution and the other as a compensating solution. By means of burettes, make additions as follows:

a) Test solution

Add 15 ml of sodium metabisulphite solution (4.9). Swirl, boil for one minute to expel sulphur dioxide. Without removing from the source of heat add exactly 50 ml of molybdate reagent (4.12). Bring just to the boil and place in a water-bath at 85°C to 90 °C for 20 min or bring to the boil and maintain for 4 min.

Remove from the source of heat and cool quickly to ambient temperature under cold water.

Transfer to a 100 ml volumetric flask, dilute to the mark with water and mix.

b) Compensating solution

Add 15 ml sodium metabisulphite solution (4.9). Swirl, then boil for one minute to expel sulphur dioxide. Without removing from the source of heat add 50 ml of sulphuric acid (4.8).

Remove from the source of heat and allow to cool to ambient temperature.

Transfer to a 100 ml volumetric flask. Dilute to the mark with water and mix.

7.3.3 Spectrophotometric measurements

Set the spectrophotometer to zero absorbance in relation to water. Carry out spectrophotometric measurements on the blank, the test solution, and the compensating solution at the maximum of the absorption curve, either at a wavelength of about 680 nm in 2 cm optical cells or at a wavelength of about 825 nm for 1 cm optical cells.

Substract the absorbance of the compensating solution from that of the test solution. Convert the net readings for the test portion solution and for the blank test solution to (µg) micrograms of phosphorus by reference to the calibration graph (7.4.4).

7.4 Establishment of the calibration graph

7.4.1 Preparation of calibration solutions

In a series of six wide-necked 250 ml conical flasks introduce phosphorus standard solution (4.13) as indicated in the following table: