SLOVENSKI STANDARD
SIST EN 10184:1997

01-maj-1997

Kemična analiza železovih zlitin - Določanje fosforja v jeklih in železovih litinah - Spektrofotometrična metoda

Chemical analysis of ferrous materials - Determination of phosphorus in steels and irons - Spectrophotometric method

Chemische Analyse von Eisenwerkstoffen - Bestimmung von Phosphor in Stahl und Eisen - Spektralphotometrisches Verfahren

Analyse chimique des matériaux sidérurgiques - Dosage du phosphore dans les aciers et fontes - Méthode spectrophotométrique

Ta slovenski standard je istoveten z: **EN 10184:1989**

ICS:

77.040.30 Kemijska analiza kovin Chemical analysis of metals

SIST EN 10184:1997 **en**

EUROPEAN STANDARD EN 10 184

NORME EUROPEENNE

EUROPAISCHE NORM May 1989

UDC 669.1:543.422.4:546.18

Key words: Iron- and steel products, steels, cast iron, chemical analysis, determination of content, phosphorus, spectrophotometric analysis, phospho-vanado-molybdate, molecular absorption spectrophotometry.

English version

Chemical analysis of ferrous materials. Determination of phosphorus in steels and irons. Spectrophotometric method

Analyse chimique des matériaux sidérurgiques. Dosage du phosphore dans les aciers et fontes. Méthode spectrophotométrique

Chemische Analyse von Eisenwerkstoffen. Bestimmung von Phosphor in Stahlen und Eisen. Spektralphotometrisches Verfahren

This European Standard was accepted by CEN on 1989-05-15.
CEN members are bound to comply with the requirements of the CEN/CENELEC Common Rules which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Central Secretariat or to any CEN member.

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

CEN members are the national standards organizations of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxemburg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

Central Secretariat: rue Bréderode 2, B-1000 Brussels

Ref. No. EN 10 184:1989 E

Brief History

This European Standard takes over the contents of EURONORM 184-87 "Chemical analysis of ferrous materials - Determination of phosporus in steels and irons - Spectrophotometric method" prepared by ECISS/TC 20 "Methods of chemical analysis" the secretariat of which is allocated to the Dansk Standardiseringsrad (DS).

It was 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.

According to the Common CEN/CENELEC Rules, following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxemburg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Chemical analysis of ferrous materials Determination of phosphorus in steels and irons Spectrophotometric method

FOREWORD

This European Standard specifies two methods for the spectrophotometric determination of phosphorus.

The first method is applicable to steels and irons and corresponds to International Standard ISO 2732-1984.

The second method is applicable to non-alloyed steels and irons.

METHOD 1

Steel and cast iron – Determination of phosphorus content – Phosphovanadomolybdate spectrophotometric method [1]

CONTENTS

1 SCOPE AND FIELD OF APPLICATION

This European Standard specified a spectrophotometric method for the determination of phosphorus in steel and cast iron.

The method is applicable to phosphorus contents between 0.005 and 1.5 % (m/m), provided that tungsten, niobium, tantalum and zirconium contents are not higher than 1 % (m/m) for each of these four elements and titanium content is not higher than 2 % (m/m).

2 REFERENCE

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

3 PRINCIPLE

Dissolution of a test portion in an oxidizing acid mixture.

Conversion of phosphorus to phosphovanadomolybdate in perchloric-nitric acid solution.

Extraction of phosphovanadomolybdate into 4-methyl-2-pentanone with citric acid present to complex arsenic.

Spectrophotometric measurement at a wavelength of about 425 nm.

(1) The text of this European Standard corresponds with the text of ISO 2732 – 1984.

4 REAGENTS

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

Verify by blank tests 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.

4.1 Pure iron, containing 0.001 % (m/m) or less of phosphorus.

4.2 Hydrochloric acid, ρ about 1.19 g/ml.

4.3 Nitric acid, ρ about 1.40 g/ml.

4.4 Nitric acid, diluted 1 + 4.

4.5 Perchloric acid, ρ about 1.54 g/ml, with known low phosphorus content.

Note: Perchloric acid (ρ about 1.67 g/ml) may also be used. 100 ml of perchloric acid (ρ about 1.54 g/ml) is equivalent to 79 ml of perchloric acid (ρ about 1.67 g/ml).

4.6 Citric acid, solution

Dissolve 500 g of citric acid monohydrate ($H_8C_6O_7 \cdot H_2O$) in water, dilute to 1 000 ml and mix.

4.7 4-Methyl-2-pentanone (isobutyl methyl ketone).

4.8 Hexaammonium heptamolybdate, solution.

Dissolve 15 g of hexaammonium heptamolybdate tetrahydrate [$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$] in water, dilute to 100 ml and mix.

4.9 Ammonium metavanadate, solution.

Dissolve 2.5 g of ammonium metavanadate (NH_4VO_3) in water, dilute to 1 000 ml and mix.

4.10 Boron fluoride, solution.

Dissolve 40 g of boric acid (H_3BO_3) in 300 ml of water in a plastic beaker, add 100 ml of hydrofluoric acid (ρ about 1.13 g/ml, dilute to 1 000 ml and mix.

Keep the solution in a plastic bottle.

4.11 Potassium permanganate, 10 g/l solution.

4.12 Sodium nitrite, 50 g/l solution.

4.13 Phosphorus, standard solution.

4.13.1 Phosphorus, 0.1 g/l stock solution.

Weigh, to the nearest 0.0001 g, 0.439 3 g of potassium dihydrogen orthophosphate (KH_2PO_4), previously dried to constant mass at 105 °C and cooled in a desiccator.

Dissolve in water, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0.1 mg of P.

4.13.2 Phosphorus, 0.01 g/l standard solution.

Transfer 100.0 ml of the stock solution (4.13.1) to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

This solution should be prepared immediately before use.

1 ml of this solution contains 0.01 mg of P.

5 APPARATUS

Ordinary laboratory apparatus and spectrophotometer.

6 SAMPLING

Carry out sampling in accordance with Euronorm 18.

7 PROCEDURE

Warning: Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic material in general.

7.1 Test portion

Weigh, to the nearest 0.001 g, approximately 0.5 g (m) 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 the reagents.

7.3 Determination

7.3.1 Preparation of test solution

7.3.1.1 For phosphorus content equal to or less than 0.08 % (m/m)

Introduce the test portion (7.1) into a 125 ml conical beaker. Add 5 ml of the nitric acid (4.3) and 3 ml of the hydrochloric acid (4.2), cover the beaker with a watch-glass and heat until solvent action ceases (see *Note* below).

Add 10 ml of the perchloric acid (4.5) and evaporate to fuming. Continue fuming for 5 to 10 minutes at such a temperature that a steady reflux of white perchloric acid fumes on the walls of the beaker is maintained (see 9.1).

Cool, add 25 ml of the nitric acid (4.4) and a few glass beads and then boil for 1 to 2 minutes (see 9.2).

Add 5 ml of the potassium permanganate solution (4.11), boil for 2 minutes, then add 10 ml of the sodium nitrite solution (4.12) and boil until free of nitrous fumes.

Note: For highly alloyed samples which do not dissolve readily in this acid mixture, further 5 ml additions of the hydrochloric acid (4.2) may be made up to a maximum of 20 ml.

7.3.1.2 For phosphorus content greater than 0.08 % (m/m)

Carry out the same procedure as specified in the first paragraph of 7.3.1.1.

Dilute to about 100 ml with water, and filter if necessary to remove graphite.

Cool, transfer quantitatively into a 200 ml one-mark volumetric flask, dilute to the mark and mix.

Take a suitable volume (V) of the solution, containing not more than 0.4 mg of phosphorus.

Proceed as specified in 7.3.1.1 from the second paragraph beginning at 'Add 10 ml of the perchloric acid (4.5) ...' to the end of 7.3.1.1.

7.3.2 Colour development

Cool to about 20°C.

Add 10.0 ml of the ammonium metavanadate solution (4.9) and 15.0 ml of the hexaammonium heptamolybdate solution (4.8) and then allow to stand at a temperature between 18 and 25°C for a minimum of 7 minutes.

Transfer the solution to a 250 ml separating funnel marked at 100 ml, dilute to the mark with water and mix. Add 10 ml of the citric acid solution (4.6), mix and immediately add 40.0 ml of the 4-methyl-2-pentanone (4.7), and shake the funnel for 30 seconds.

Allow the two layers to separate and discard the lower (aqueous) layer.

Dry the inside of the stem of the separating funnel with a small piece of filter paper. Filter the 4-methyl-2-pentanone layer through a dry rapid paper into a small dry beaker.

7.3.3 Spectrophotometric measurement

Carry out the spectrophotometric measurements at 20 ± 1°C in cells of 2 cm optical path at a wavelength of about 425 nm after having adjusted the spectrophotometer (Clause 5) to zero against the 4-methyl-2-pentanone (4.7).

Correct the absorbance with the blank test solution.

7.4 Establishment of the calibration graph

7.4.1 Preparation of calibration solutions

Introduce into a series of nine 125 ml conical beakers 0.5 g of the pure iron (4.1) and then respectively the volumes of the phosphorus standard solution (4.13.2) indicated in the following table:

Volume of phosphorus standard solution (4.13.2)	Corresponding mass of phosphorus
ml	mg
0	0
5.0	0.05
10.0	0.10
15.0	0.15
20.0	0.20
25.0	0.25
30.0	0.30
35.0	0.35
40.0	0.40

Proceed as specified in 7.3.1.1 from the first paragraph beginning at 'Add 5 ml of the nitric acid (4.3), 5 ml of the hydrochloric acid (4.2)...' to the end of 7.3.2, but omitting 7.3.1.2.

7.4.2 Spectrophotometric measurement

Carry out spectrophotometric measurements of each solution in cells of 2 cm optical pathlength at a wavelength of about 425 nm after having adjusted the spectrophotometer (Clause 5) to zero absorbance against the zero term of the calibration solutions (7.4.1).

7.4.3 Plotting of the calibration graph and calculation of the angular coefficient *a*

Plot the absorbance against the known mass of phosphorus, in milligrams in 40 ml of 4-methyl-2-pentanone.

Calculate the angular coefficient *a* from the slope of the calibration graph, if it is a straight line.

8 EXPRESSION OF RESULTS

The phosphorus (P) content, as a percentage by mass, is obtained in accordance with the calibration graph, or calculated from the following formula:

for phosphorus content equal to or less than 0.08 % (m/m):

$$\frac{A}{a} \cdot \frac{1}{m} \cdot \frac{1}{10^3} \cdot 100 = \frac{A}{10\,a\,m}$$

or

for phosphorus content greater than 0.08 % (m/m):

$$\frac{200}{V} \cdot \frac{A}{a} \cdot \frac{1}{m} \cdot \frac{1}{10^3} \cdot 100 = \frac{20\,A}{V\,a\,m}$$

where

a is the angular coefficient or absorbance of a solution containing 1 mg of phosphorus in 40 ml of 4-methyl-2-pentanone with an optical pathlength of 2 cm.

A is the absorbance of the test solution corrected by the absorbance of its blank test.

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

V is the volume, in millilitres, taken from the test solution for phosphorus content greater than 0.08 % (m/m).

9 NOTES

9.1 For samples containing more than 25 % (m/m) of chromium, remove the chromium by volatilization as follows:

To the fuming solution, with the chromium fully oxidized, add 5 ml of the hydrochloric acid (4.2), then continue fuming until the remaining chromium is again fully oxidized.

Repeat the treatment with hydrochloric acid followed by further fuming to reoxidize the small amount of residual chromium.

9.2 For samples containing titanium, zirconium, niobium or tantalum contents as specified in Clause 1, after evaporation of perchloric acid, add 20 ml of the boron fluoride solution (4.10) in addition to the nitric acid (4.4).

10 TEST REPORT

The test report shall include the following information:

(a) the method used by reference to this European standard;

(b) the results, and the form in which they are expressed;

(c) any unusual features noted during the determination;

(d) any operation not specified in this European Standard or in the European Standard to which reference is made, or any optional operation which may have influenced the results.