17

International Standard 2732

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Steel and cast iron — Determination of phosphorus content — Phosphovanadomolybdate spectrophotometric method

Aciers et fontes — Dosage du phosphore — Méthode spectrophotométrique au phosphovanadomolybdate

Second edition — 1984-08-15

UDC 669.13/.14 : 543.42 : 546.18 **Ref. No. ISO 2732-1984 (E)**

Descriptors : steels, cast iron, chemical analysis, determination of content, phosphorus, spectrophotometric analysis.

Price based on 3 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2732 was developed by Technical Committee ISO/TC 17, *Steel*, and was circulated to the member bodies in June 1983.

It has been approved by the member bodies of the following countries :

Australia
Austria
Belgium
Brazil
Bulgaria
China
Czechoslovakia
Finland
Germany, F. R.
Hungary
India
Iran
Italy
Japan
Kenya
Korea, Dem. P. Rep. of
Korea, Rep. of
Mexico
Netherlands
New Zealand
Norway
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Tanzania
Turkey
United Kingdom
Venezuela
Yugoslavia

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Sweden
USSR

This second edition cancels and replaces the first edition (i.e. ISO 2732-1973).

Printed in Switzerland

INTERNATIONAL STANDARD

ISO 2732-1984 (E)

Steel and cast iron — Determination of phosphorus content — Phosphovanadomolybdate spectrophotometric method

1 Scope and field of application

This International Standard specifies 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

ISO/R 377, *Selection and preparation of samples and test pieces for wrought steel.*

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.

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 ($H_8C_6O_7.H_2O$) 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 $[(NH_4)_6Mo_7O_{24}.4H_2O]$ 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 plastics beaker, add 100 ml of hydrofluoric acid (ϱ 1,13 g/ml), dilute to 1 000 ml and mix.

Keep the solution in a plastics 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 0,000 1 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 ISO/R 377, or appropriate national standards for cast iron.

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 5 ml of the hydrochloric acid (4.2), cover the beaker with a watch-glass and heat until solvent action ceases (see the note).

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

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

Add 5 ml of the potassium permanganate solution (4.11), boil for 2 min, 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 min.

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 s.

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 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 formulae :

for phosphorus content equal to or less than 0,08 % (*m/m*) :

$$\frac{A}{a} \times \frac{1}{m} \times \frac{1}{10^3} \times 100$$

$$= \frac{A}{10\,a\,m}$$

or

for phosphorus content greater than 0,08 % (*m/m*) :

$$\frac{200}{V} \times \frac{A}{a} \times \frac{1}{m} \times \frac{1}{10^3} \times 100$$

$$= \frac{20\,A}{V\,a\,m}$$

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 International 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 International Standard or in the International Standard to which reference is made, or any optional operation which may have influenced the results.