INTERNATIONAL STANDARD ISO 2732

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Steels and cast iron – Determination of phosphorus – Spectrophotometric method

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FOREWORD

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INTERNATIONAL STANDARD **ISO 2732-1973 (E)**

Steels and cast iron — Determination of phosphorus — Spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method for the determination of phosphorus in steels and cast iron.

The method is applicable to phosphorus contents from 0,005 to 1,5 %, provided that the tungsten, niobium, tantalum and titanium contents are not higher than 1 % for each of these four elements.

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 the phosphorus to phosphovanadomolybdate in perchloric-nitric acid solution. Extraction of the phosphovanadomolybdate into isobutyl methyl ketone with citric acid present to complex arsenic. Spectrophotometric measurement at a wavelength of about 425 nm.

4 REAGENTS

Demineralized water is to be used with the present method.

Verify by blank tests that the relevant reagents are free from phosphorus. Wherever necessary, the results must be corrected accordingly. Grades giving high blank values are unsuitable and should be discarded.

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml.

4.2 Iron, of high purity, containing 0,001 % or less of phosphorus.

4.3 Isobutyl methyl ketone (4-methylpentan-2-one).

4.4 Nitric acid, ρ approximately 1,40 g/ml.

4.5 Perchloric acid, ρ 1,54 g/ml, with known low phosphorus content.

NOTE — 70 % perchloric acid (ρ approximately 1,67 g/ml) may also be used : 1 000 ml of perchloric acid (ρ 1,67 g/ml) is equivalent to 1 270 ml of perchloric acid (ρ 1,54 g/ml).

4.6 Ammonium molybdate solution, 150 g/l.

Dissolve in water 15 g of ammonium molybdate $[(NH_4)_6Mo_7O_{24}.4H_2O]$ (analytical reagent quality), dilute to 100 ml and mix.

4.7 Ammonium vanadate solution, 2,5 g/l.

Dissolve 2,5 g of ammonium metavanadate (NH_4VO_3) (analytical reagent quality) in water, dilute to 1 l and mix.

4.8 Citric acid solution, 500 g/l.

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

4.9 Nitric acid (20 % *V/V*).

Dilute 200 ml of nitric acid (ρ approximately 1,40 g/ml) to 1 l and mix.

4.10 Potassium permanganate solution, 10 g/l.

Dissolve 1 g of potassium permanganate in water, dilute to 100 ml and mix.

4.11 Sodium nitrite solution, 50 g/l.

Dissolve 5 g of sodium nitrite in water, dilute to 100 ml and mix.

4.12 Phosphorus standard solution.

Dissolve in water 0,439 3 g of potassium dihydrogen orthophosphate ($KH_2\ PO_4$) (analytical reagent grade), previously dried to constant mass at 105 °C. Cool, transfer to a 1 l volumetric flask, dilute to the mark and mix. Transfer 100 ml of this solution to a 1 l volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0,01 mg of phosphorus.

5 APPARATUS

Ordinary laboratory equipment, and

5.1 Spectrophotometer.

6 SAMPLING

Carry out sampling in accordance with ISO/R 377, or the appropriate national standards for cast iron.

7 PROCEDURE

7.1 Test portion

0,5 ± 0,001 g.

7.2 Blank test

Parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all the reagents.

7.3 Plotting of calibration curve

Into a series of nine 125 ml conical beakers, introduce 0,5 g of high purity iron (4.2), then respectively the volumes of phosphorus standard solution (4.12) indicated in the following table :

Volume of phosphorus standard solution (4.12)	Corresponding mass of phosphorus
ml	mg
0	0
5,0	0,05
10,0	0,1
15,0	0,15
20,0	0,2
25,0	0,25
30,0	0,3
35,0	0,35
40,0	0,4

Add a mixture of 5 ml of nitric acid (4.4) and 5 ml of hydrochloric acid (4.1), cover the beaker and heat until solvent action ceases. Add 10 ml of perchloric acid (4.5) (see Note 9.1) and evaporate to fuming.

Continue fuming for 5 to 10 min at such a temperature that a steady reflux of acid on the walls of the beaker is maintained.

Cool, add 25 ml of nitric acid (4.9) and a few glass beads, then boil for 1 to 2 min. Add 5 ml of potassium permanganate solution (4.10), boil for 2 min, then add 10 ml of sodium nitrite solution (4.11) and boil until free of nitrous fumes. Continue as in 7.5.

Carry out the spectrophotometric measurement of each solution in the range with reference to that containing no added phosphorus.

Plot the calibration curve or calculate its angular coefficient if it is a straight line.

7.4 Preparation of sample solution

Introduce the test portion (7.1) into a 125 ml conical beaker. Add a mixture of 5 ml of nitric acid (4.4) and 5 ml of hydrochloric acid (4.1), cover the beaker and heat until solvent action ceases (see Notes 9.3 and 9.4). Add 10 ml of perchloric acid (4.5) (see Note 9.1) and evaporate to fuming. Continue fuming for 5 to 10 min at such a temperature that a steady reflux of acid on the walls of the beaker is maintained (see Note 9.5).

Cool, add 25 ml of nitric acid (4.9) and a few glass beads, then boil for 1 to 2 min.

Add 5 ml of potassium permanganate solution (4.10), boil for 2 min, then add 10 ml of sodium nitrite solution (4.11) and boil until free of nitrous fumes.

7.5 Determination

Cool to about 20 °C, add 10 ml of ammonium vanadate solution (4.7) (see Note 9.2) and 15 ml of ammonium molybdate solution (4.6), then allow to stand for a minimum of 7 min (see Note 9.6).

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 citric acid solution (4.8), mix and immediately add 40 ml of isobutyl methyl ketone (4.3) 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 ketone layer through a dry rapid paper into a small dry beaker.

Carry out spectrophotometric measurements at 20 ± 1 °C in cells of 2 cm optical path at a wavelength of approximately 425 nm without going beyond an absorbance of 1.

8 EXPRESSION OF RESULTS

8.1 Conversion of spectrophotometric measurements

Correct the spectrophotometric readings for reagent blank determined as in 7.2 and convert to percentage phosphorus by reference to the appropriate calibration curve.

8.2 Accuracy of determination

The limits of accuracy of this method will be given later.

9 NOTES ON PROCEDURE

9.1 Perchloric acid fumes attack the wood of fume cupboards. All exposed parts should be washed at regular intervals. Wooden benches should not be used when working with perchloric acid. These fumes may cause explosions in the presence of ammonia or nitric acid fumes.

9.2 It is recommended that this and all subsequent reagent additions be made by means of a pipette or burette.

9.3 For highly alloyed samples which do not dissolve readily in this acid mixture, further 5 ml additions of hydrochloric acid (4.1) may be made up to a maximum of 20 ml.

9.4 For phosphorus contents greater than 0,08 %, dilute, filter if necessary to remove graphite, cool and dilute to 200 ml in a volumetric flask. Take a suitable aliquot containing not more than 0,4 mg of phosphorus.

9.5 For samples containing more than 25 % of chromium, remove the chromium by volatilization as follows :

To the fuming solution, with the chromium fully oxidized, add 5 ml of hydrochloric acid (4.1), 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.

9.6 At a temperature of 20 °C the colour development is complete after standing for 7 min but may be retarded if titanium or zirconium is present.

Complete colour development is obtained for samples containing up to 1 % of titanium and 0,5 % of zirconium by increasing the standing time to 1 h.

10 TEST REPORT

The test report shall include the following particulars :

a) reference to this International Standard;

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

c) any characteristics noted during the determination and also any operations not specified in this International Standard which may have affected the results.