BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

Draft Indian Standard

METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 5 DETERMINATION OF PHOSPHORUS (0.01 TO 0.50 PERCENT) BY ALKALIMETRIC METHOD

[*First Revision* of IS 12308 (Part 5)]

ICS 77.080.10

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34	Last date of comments **16 March 2023**

FOREWORD

This draft Indian Standard (Part 5) (First Revision) subject to its finalization, is to be adopted by the Bureau of Indian Standards on recommendation of the Methods of Chemical analysis of Metals Sectional Committee and approval of the Metallurgical Engineering Division Council.

Chemical analysis of cast iron and pig iron was covered in IS 228 : 1959 'Methods of chemical analysis of pig iron, cast iron and plain carbon and low alloy steels (*revised*)'. During its second revision it was decided that a comprehensive series should be prepared for chemical analysis of all types of steels and the other covering the chemical analysis of cast iron and pig iron. Accordingly IS 228 on revision was published in several parts covering chemical analysis of various steels only and a separate series of standards under IS 12308 is being published for chemical analysis of cast iron and pig iron.

This standard was first published in 1991 in different parts covering methods for chemical analysis of cast iron and pig iron. This standard (Part 5) covers determination of phosphorus (0.01 to 0.50 percent) by alkalimetric method. The method given in this part for determination of phosphorus had been updated on the basis of experience gained over the period.

The other parts in the series are:

Part 1	Determination of total carbon by thermal conductivity method
Part 2	Determination of sulphur by iodimetric titration method
Part 3	Determination of manganese by periodate spectrophotometric method
Part 4	Determination of total carbon, graphitic carbon and combined carbon by gravimetric method
Part 6	Determination of Silicon (for Silicon 0.1 to 6.0 percent)
Part 7	Determination of nickel by dimethylglyoxime (Gravimetric) method (for nickel 0.5 to 36 percent)

Part 8	Determination of chromium by persulphate oxidation method (for chromium 0.1 to 28 percent)
Part 9	Determination of molybdenum by thiocyanate (Spectrophotometric) method (for molybdenum 0.1 to 1.0 percent)
Part 10	Determination of manganese (up to 7.0 percent) by arsenite (Volumetric) method
Part 11	Determination of total carbon by the direct combustion volumetric method (for carbon 1.50 to 4.50 percent)
Part 12	Determination of copper by atomic absorption spectrometric method (for copper 0.01 to 0.5 percent)
Part 13	Determination of magnesium by atomic absorption spectrometric method (for magnesium upto 0.1 percent)
Part 14	Determination of titanium by hydrogen peroxide (Spectrophotometric) method (for titanium up to 0.25 percent)

This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Draft Indian Standard

METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 5 DETERMINATION OF PHOSPHORUS (0.01 TO 0.50 PERCENT) BY ALKALIMETRIC METHOD

(First Revision)

1 SCOPE

This standard (Part 5) prescribes the method for determination of phosphorus, in the range of 0.01 to 0.50 percent, in cast iron and pig iron.

2 REFERENCE

The Indian Standards listed below contains provisions which through reference in this text, constitutes provisions of this standard. At the time of publication the edition indicated was valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No	*Title*
IS 264 : 2005	Nitric acid – specification (*third revision*)

3 SAMPLING

The sample shall be drawn and prepared as prescribed in the relevant Indian Standard**.**

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

5 METHOD

5.1 Outline of the Method

Phosphorus is converted to orthophosphoric acid and precipitated as ammonium phosphomolybdate. The precipitate is dissolved in known excess of standard sodium hydroxide solution and excess is titrated against standard nitric acid solution.

5.2 Reagents

5.2.1 *Concentrated Nitric Acid* – rd = 1.42 (conforming to IS 264).

5.2.2 *Dilute Nitric Acid* – 1 percent, 2 percent and 2:3 (*v/v*)

5.2.3 *Potassium Permanganate Solution* – 2 percent (*m/v*).

5.2.4 *Sodium Nitrite* – 2 percent (*m/v*).

5.2.5 *Concentrated Ammonium Hydroxide* – rd = 0.90.

5.2.6 *Ammonium Molybdate Solution*

Add *Solution A (see* **5.2.6.1**) slowly and with constant stirring to *Solution B* (*see* **5.2.6.2**) kept cool in a cold waterbath. Add 10 ml of ammonium phosphate solution (1 g/l) and keep the solution at least for 24 hours. Filter the solution through medium textured filter paper before use.

5.2.6.1 *Solution A*

Dissolve 100 g of molybdic acid (MoO_3, 85 percent), or 118 g of ammonium molybdate in a mixture of 145 ml of ammonium hydroxide (rd = 0.90) and 270 ml of water. Cool the solution.

5.2.6.2 *Solution B*

Add 300 ml of concentrated nitric acid to 700 ml of water and cool.

5.2.7 *Potassium Nitrate Solution* – 1 percent (*m/v*).

5.2.8 *Phenolphthalein Indicator Solution* – 0.2 percent.

Dissolve 0.2 g of phenolphthalein powder in 80 ml of rectified spirit and make up to 100 ml with the rectified spirit.

5.2.9 Standard Sodium Hydroxide Solution – 0.1 N.

Dissolve 4.5 g of sodium hydroxide in one litre of freshly boiled and cooled distilled water, and standardize against standard nitric acid (**5.2.10**).

5.2.10 *Standard Nitric Acid Solution* – 0.1 N.

Dilute 7 ml of concentrated nitric acid to one litre with freshly boiled distilled water. Standardize against sodium carbonate previously heated at 275-300 °C and cooled. 1 ml of this solution will then be equivalent to 0.003 135 g of phosphorus.

5.3 Procedure

5.3.1 Transfer 0.200 to 2.000 g of sample (containing 0.2 to 2 mg of P) to 250 ml beaker and dissolve in 30 ml of dilute nitric acid (2:3) (*see* Notes 1 and 2). Digest until dissolve. Evaporate to syrupy consistency and cool. Add 2 ml of concentrated nitric acid and 50 ml of water. Filter and wash 4 to 5 times with dilute nitric acid (1 percent). Take the clear filtrate and while boiling add potassium permanganate solution drop wise till a brown precipitate of manganese dioxide appears. Boil for another 2 minutes. Add sodium nitrite solution drop wise to clear the brown precipitate of manganese. Add another 2 to 3 drops of sodium nitrite and boil for 5 minutes to expel oxides of nitrogen. Cool the solution and to the cooled solution add ammonium hydroxide drop by drop till neutralization. Add 2 to 3 drops of nitric acid to clear the solution. Add 5 ml of nitric acid. The volume at this stage should be about 80 ml. Heat the solution to 60-80 °C and transfer to a 250 ml conical flask.

NOTES

1 If sample is not soluble in nitric acid, dissolve in mixture of nitric acid and hydrochloric acid. When sample is decomposed add perchloric acid and evaporate nearly to dryness. Add 30 ml of nitric acid and proceed.

2 If vanadium is present, add 2 to 3 ml of sodium nitrite during the removal of manganese dioxide and precipitation of phosphate is performed at 10 to 20 °C.

5.3.2 To the warm solution add 30 ml of ammonium molybdate solution. Stopper the flask, shake vigorously for a few minutes and allow to stand for half an hour. Filter the precipitate through medium textured filter paper or filter paper pulp. Wash the precipitate with dilute nitric acid solution (1 percent), once or twice. Finally wash the flask, the precipitate and the paper, with 5 ml portions of potassium nitrate Gash solution until 10 ml of the filtrate collected in a test tube does not consume more than one drop of the standard sodium hydroxide solution (**5.2.9**) in presence of a drop of phenolpthalein indicator solution.

NOTE – Generally 3 or 4 washings with potassium nitrate solution are sufficient for the precipitate to be acid free.

5.3.3 Transfer the paper and precipitate to the flask in which precipitation was carried out. Add about 25 ml of water, and a known volume of standard sodium hydroxide solution (which should be 2 to 5 ml in excess) and shake to dissolve the precipitate. Dilute to about 100 ml and titrate with standard nitric acid solution using phenolphthalein as indicator.

5.3.4 Carry out a blank determination following the same procedure as specified in **5.3.1** to **5.3.3** and using the same quantity of all reagents.

5.4 Calculation

$$\text{Phosphorus content, percent by mass} = \frac{(B - A) \times C}{D} \times 100$$

where

B = volume, in ml, of standard nitric acid solution required for the blank (**7.1**);

A = volume, in ml, of standard nitric acid solution required for the sample (*see* **5.3.3**);

C = phosphorus equivalent of 1 ml standard nitric acid solution (*see* **5.2.10**); and

D = mass, in g, of the sample taken for test.

5.5 Reproducibility

± 0.001 5 percent.