***भारतीय मानक*  IS 12308 (Part 5) : 2024**

***Indian Standard***

**ढलवाँ लोहा और कच्चा लोहा के रासायनिक विश्लेषण की पद्धतियाँ**

**भाग 5 क्षारमितीय पद्धति द्वारा (0.01 प्रतिशत से 0.50 प्रतिशत) फॉस्फोरस का निर्धारण**

 *(* पहला *पुनरीक्षण )*

**Methods for Chemical Analysis of Cast Iron and Pig Iron**

**Part 5 Determination of Phosphorus(0.01 Percent to 0.50 Percent)by Alkalimetric Method**

*( First Revision )*

ICS 77.080.10

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भारतीय मानक ब्यूरो

BUREAU OF INDIAN STANDARDS

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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 5) (First Revision) was adopted by the Bureau of Indian Standards after the finalized by the Methods of Chemical analysis of Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1991. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

This standard is published in different parts covering methods for chemical analysis of cast iron and pig iron are as listed below. This standard (Part 5) covers determination of phosphorus (0.01 percent to 0.50 percent) by alkalimetric method.

The other parts in the series are:

|  |  |
| --- | --- |
| Part 1 | Determination of total carbon by thermal conductivity method (for carbon 1.00 percent to 4.50 percent) |
| Part 2 | Determination of sulphur by iodimetric titration method by iodimetric titration after combustion (for sulphur 0.005 percent to 0.25 percent) |
| Part 3 | Determination of manganese by periodate spectrophotometric method (for manganese 0.1 percent to 2.5 percent) |
| Part 4 | Determination of total carbon, graphitic carbon and combined carbon by gravimetric method |
| Part 6 | Determination of silicon by gravimetric method (for silicon 0.1 percent to 6.0 percent) |
| Part 7 | Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel 0.5 percent 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 percent 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) |

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

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*)’.

*Indian Standard*

METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

**PART 5 DETERMINATION OF PHOSPHORUS (0.01 PERCENT 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 percent to 0.50 percent, in cast iron and pig iron.

**2 REFERENCE**

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

|  |  |
| --- | --- |
| *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* ― relative density = 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* ― relative density = 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 h. Filter the solution through medium textured filter paper before use.

**5.2.6.1** *Solution A*

Dissolve 100 g of molybdic acid (MoO3, 85 percent), or 118 g of ammonium molybdate in a mixture of 145 ml of ammonium hydroxide (relative density = 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 °C to 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 g to 2.000 g of sample (containing 0.2 mg 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 min. 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 min 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 °C to 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 ml to 3 ml of sodium nitrite during the removal of manganese dioxide and precipitation of phosphate is performed at 10 °C 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 ml 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**

Phosphorus content, percent by mass = $\frac{\left(B - A\right) × C}{D}$ × 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.

**ANNEX A**

(*Foreword*)

**COMMITTEE COMPOSITION**

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

| *Organization* |  | *Representative(s)* |
| --- | --- | --- |
| CSIR - National Metallurgical Laboratory, Jamshedpur |  | Dr Sanchita Chakravarty **(*Chairperson*)** |
| Arcelor Mittal Nippon Steel, Mumbai |  | Shri Manoj Gupta |
|  Shri Kirit Tailor (*Alternate*) |
| Bhabha Atomic Research Centre, Mumbai |  | Miss Sanjukta A. Kumar |
|  | Shri M. V. Rana (*Alternate*) |
| CSIR - National Metallurgical Laboratory, Jamshedpur |  | Dr Ashok K. Mohanty (*Alternate*) |
| Defence Metallurgical Research Laboratory, Ministry of Defence, Hyderabad |  | Shri S. S. Kalyan Kamal |
| Directorate General of Quality Assurance, Ministry of Defence, New Delhi |  | Shri Kesavamoorthy M. |
|  | Shri E. Suman Kumar (*Alternate*) |
| Geological Survey of India, New Delhi |  | Shri Nitin Purushottam |
|  |  Shrimati Sanjukta Dey Pal (*Alternate*) |
| Hindalco Industries Limited, Mumbai |  | Shri Krishanu Mahapatra |
|  | Shri Ashutosh Acharya (*Alternate*) |
| Indian Metals and Ferro Alloys Limited, Bhubaneswar |  | Shri Dinesh Kumar Mohanty |
| Jawaharlal Nehru Aluminium Research Development and Design Centre, Nagpur |  | Dr Upendra Singh  |
| JSW Steel Limited, Mumbai |  | Shri Kotrabasavaraju |
|  | Shri Marulasiddesha U. M. (*Alternate*) |
| National Aluminium Company Limited, Bhubaneswar |  | Shrimati Sukla Nandi |
|  | Shri Debananda Bhattacharyya (*Alternate*) |
| National Mineral Development Corporation, Hyderabad |  | Dr Saroj Kumar Sahu |
|  |  Shri Ashish Shrivastava (*Alternate*) |
| National Test House, Kolkata |  | Dr Rajeev Kumar Upadhyay |
|  | Shri Akbar H. (*Alternate*) |
| Research Designs and Standards Organization (RDSO), Lucknow |  | Shri Sandeep |
| Shriram Institute for Industrial Research, Delhi |  | Dr Laxmi Rawat |
|  | Shri Puneet Kapoor (*Alternate*) |
| Steel Authority of India Limited - Salem Steel Plant, Salem |  | Shri L. Sivakumar |
|  | Shri Vivekanandhan G. (*Alternate*) |
| Tata Steel Limited, Kolkata |  | Dr Jatin Mohapatra |
|  |  Dr Ravikrishna Chatti (*Alternate*) |
| TRL Krosaki Refractories Limited, Belpahar |  | Shri S. K. Subudhi |
| BIS Directorate General |  | Shri Sanjiv Maini, Scientist ‘F’/Senior Director and Head (Metallurgical Engineering) [Representing Director General (*Ex-officio*)] |

*Member Secretary*

Shri Ashish Prabhakar Wakle

Scientist ‘D’/Joint Director

(Metallurgical Engineering), BIS