भारतीय मानक **IS 228 (Part 3) : 2024**

Indian Standard

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(चौथा पुनरीक्षण)

Methods for Chemical Analysis of Steels

Part 3 Determination of Phosphorus by Alkalimetric Method

(Fourth Revision)

ICS 77.080.20

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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 3) (Fourth Revision) was adopted by the Bureau of Indian Standards after the draft 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 1952 and subsequently revised in 1959, 1972 and 1987.This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

This part covers method for the determination of phosphorus content of plain carbon steel and alloy steels by alkalimetric method. The other parts of this series are:

Part 1	Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent)
Part 2	Determination of manganese in Plain-carbon and low alloy steels by arsenite method
Part 4	Determination of total carbon by gravimetric method (for carbon ≥ 0.1 percent)
Part 5	Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel ≥ 0.1 percent)
Part 6	Determination of chromium by persulphate oxidation method (for chromium ≥ 0.1 percent)
Part 7	Determination of molybdenum by alpha-benzoinoxime method in alloy steels (for molybdenum > 1 percent and not containing tungsten)
Part 8	Determination of silicon by the gravimetric method (for silicon 0.05 to 5.00 percent)
Part 9	Determination of sulphur by evolution method (for sulphur 0.01 to 0.25 percent)
Part 10	Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum 0.01 to 1.5 percent)
Part 11	Determination of total silicon by reduced molybdosilicate spectrophotometric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent)
Part 12	Determination of manganese by periodate spectrophotometric method in plain carbon, low alloy and high alloy steels (for manganese 0.01 to 5.0 percent)
Part 13	Determination of arsenic
Part 14	Determination of carbon by thermal conductivity method (for carbon 0.005 to 2.000 percent)
Part 15	Determination of copper by thiosulphate iodide method (for copper 0.05 to 5 percent)
Part 16	Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent)
Part 17	Determination of nitrogen by thermal conductivity method (for nitrogen up to 0.04 percent)
Part 18	Determination of oxygen by instrumental method (for oxygen 0.01 to 0.100 0 percent)
Part 19	Determination of nitrogen by steam distillation method (for nitrogen 0.02 to 0.50 percent)
Part 20	Determination of total carbon and sulfur content — Infrared absorption method after combustion in an induction furnace (routine method)
Part 21	Determination of copper by spectrometric method (for copper 0.02 to 0.5 percent)
Part 22	Determination of total hydrogen in steel by thermal conductivity method (hydrogen 0.1 ppm to 50 ppm)
Part 23	Determination of total nitrogen in steel by optical emission spectrometer (nitrogen 0.002 to 1.0 percent)
Part 24	Determination of nitrogen in steel by inert gas fusion — Thermal conductivity method (nitrogen 0.001 to 0.2 percent)

(*Continued on third cover*)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 3 DETERMINATION OF PHOSPHORUS BY ALKALIMETRIC METHOD

(Fourth Revision)

1 SCOPE

This standard (Part 3) covers method for determination of phosphorus content of plain carbon steel and alloy steels by alkalimetric method.

2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute provision of this standard. At the time of the publication, the editions indicated below were valid. All the 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 these standards:

IS No.	*Title*
IS 264 : 2005	Nitric acid — Specification (*third revision*)
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)

3 SAMPLING

The samples 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 (*see* IS 1070) shall be employed in the test.

5 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 the excess is titrated against standard nitric acid solution.

5.1 Reagents

5.1.1 *Dilute Nitric Acid* — 1 : 1, 1 : 3, 1 : 5 and 2 : 98 (*v/v*)

5.1.2 *Potassium Permanganate Solution*

Dissolve 2 g of potassium permanganate in 100 ml of water.

5.1.3 *Sodium Nitrite Solution* — 5 percent (*m/v*)

5.1.4 *Concentrated Ammonium Hydroxide* — relative density 0.90

5.1.5 *Concentrated Nitric Acid* — relative density 1.42 (conforming to IS 264)

5.1.6 *Ammonium Molybdate Solution*

Add Solution A (*see* 5.1.6.1) slowly and with constant stirring to Solution B (*see* 5.1.6.2) 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 Whatman filter paper No. 40 before use.

5.1.6.1 *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.

5.1.6.2 *Solution* B

Add 490 ml of concentrated nitric acid to 1 150 ml of water and cool.

5.1.7 *Potassium Nitrate Solution* — 1 percent (*m/v*)

5.1.8 *Phenolphthalein Solution* — 1 percent

Dissolve 1 g of powder phenolphthalein in 100 ml of rectified spirit or methyl alcohol.

5.1.9 *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 (*see* 5.1.10).

5.1.10 *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 ignited at 350 °C and cooled.

5.1.11 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*) and 2 percent (*v/v*)

5.1.12 *Hydrofluoric Acid* — 40 percent

5.1.13 *Perchloric Acid* — 70 percent

5.1.14 *Sodium Carbonate* — 5 percent (*m/v*)

5.1.15 *Ferrous Sulphate Solution*

Dissolve 100 g of ferrous sulphate crystals ($FeSO_4.7H_2O$) in one litre of dilute sulphuric acid (5 : 95).

5.1.16 *Dilute Ammonium Hydroxide* — 1 : 1, 1 : 20 (*v/v*)

5.1.17 *Sulphurous Acid*

Saturate water with sulphur dioxide gas.

5.1.18 *Concentrated Hydrochloric Acid* — relative density 1.16 (conforming to IS 265)

5.1.19 *Dilute Hydrobromic Acid* — 1 : 4 (*v/v*)

6 PROCEDURE FOR PLAIN CARBON STEEL AND LOW ALLOY STEEL WITHOUT VANADIUM

6.1 Transfer 2 g of the sample in a 250 ml conical flask. Add 30 ml of dilute nitric acid (1 : 1) till the vigorous reaction subsides. Keep the flask at low heat till it dissolves.

6.2 Add 5 ml to 10 ml of potassium permanganate solution and boil for few minutes. If manganese dioxide does not precipitate add further permanganate solution till a precipitate of manganese dioxide appears and boil for few minutes. Add sodium nitrite solution dropwise till the brown precipitate is dissolved. Boil to expel oxides of nitrogen.

6.3 Add ammonium hydroxide till brown precipitate of ferric hydroxide appears. Dissolve the precipitate adding dilute nitric acid (1 : 1) drop wise and add 2 ml to 3 ml of concentrated nitric acid.

NOTE — Quantity of nitric acid added at this stage will depend upon the actual volume of the solution. The analyst will ensure that the acidity of the solution before precipitation of phosphomolybdate complex is maintained at 5 percent to 10 percent.

6.4 Warm the solution to about 60 °C to 80 °C and add 50 ml of ammonium molybdate solution, stopper the flask, shake vigorously for a few minutes and allow to stand for half an hour.

6.5 Filter the precipitate through a filter paper pulp pad by suction. Wash the flask, precipitate and filter pad twice with 5 ml portions of dilute nitric acid (2 : 98) and five times with 5 ml portions of potassium nitrate. Continue washing of the precipitate and filter pad with potassium nitrate solution until the washings are acid free (10 ml portion of the washings in presence of phenolphthalein should turn pink on adding one drop of 0.1 N sodium hydroxide solution).

6.6 Transfer the pulp along with the precipitate to the flask. Add about 25 ml of water, few drops of phenolphthalein and a known volume of sodium hydroxide solution (which should be 1 ml to 2 ml in excess) and shake to dissolve the precipitate. Dilute to about 100 ml and titrate with standard nitric acid solution to the disappearance of pink colour.

7 PROCEDURE FOR ALLOY STEELS

7.1 Stainless Steel, High Chromium, Nickel Chromium and Similar Alloy Steel without Tungsten or Vanadium

Transfer 2 g of the sample in a 400 ml beaker. Add 50 ml of a 1 : 1 mixture of concentrated hydrochloric acid and nitric acid and a few drops of hydrofluoric acid. Place the beaker at low heat until the reaction subsides and then add 15 ml of perchloric acid. Evaporate to copious fumes and fume for a few minutes. Cool, take up with 40 ml of water and 10 ml of concentrated nitric acid, boil for few minutes, filter through paper pulp and wash with hot dilute nitric acid (1 : 5). Collect the filtrate in 500 ml conical flask and proceed as in 6.2 to 6.6.

7.2 Steel Containing High Silicon, Titanium or Zirconium

7.2.1 Follow the procedure given in 7.1 up to filtration and proceed as follows.

7.2.2 Collect the filtrate in a 500 ml conical flask and preserve it. Take the precipitate and pulp (as in 7.1) in a platinum crucible and ignite, fume with 1 ml hydrofluoric acid and a few drops of concentrated nitric acid at low temperature. Fuse the residue with sodium carbonate. Dissolve the melt in water, filter through paper pulp and wash with sodium carbonate solution. Acidify the filtrate with concentrated nitric acid and add to the main filtrate. Proceed further as in 6.2 to 6.6.

7.3 Chromium Vanadium Steels or other Steels Containing Vanadium but no Tungsten

7.3.1 Proceed as in 7.1 when the solution is ready for the precipitation of phosphomolybdate complex.

7.3.2 Cool the solution to 10 ºC to 20 ºC, add 5 ml of ferrous sulphate solution and 2 to 3 drops of sulphurous acid and shake to mix well. Add 50 ml of ammonium molybdate solution, stopper the flask, shake vigorously for a few minutes and allow to stand overnight. Proceed further as in 6.5 to 6.6.

7.4 High Speed Steels or Other Steels Containing Tungsten with or without Vanadium

7.4.1 Transfer 2 g of the sample to a 400 ml beaker. Decompose the sample in 60 ml of dilute nitric acid (1 : 3) and a few drops of hydrofluoric acid, if required. Add 20 ml concentrated hydrochloric acid 60 ml concentrated nitric acid and few drops of hydrofluoric acid and evaporate to dryness. Digest with 20 ml of concentrated hydrochloric acid, dilute to 50 ml with water and boil. Filter through a paper pulp as far as possible by decantation, keeping back the precipitate of tungstic acid in the beaker. Wash twice with hot dilute hydrochloric acid (2 percent). Collect the filtrate in a 500 ml conical flask and evaporate to a small volume. Add 30 ml concentrated nitric acid, and evaporate to syrupy liquid. Add 5 ml concentrated nitric acid, dilute to about 25 ml and boil for a few minutes.

7.4.2 To recover phosphorus from the precipitate of tungstic acid, dissolve the precipitate in the beaker with minimum quantity of dilute ammonium hydroxide (1 : 1) and pour it through the filter pad collecting the solution in a beaker, wash the beaker and the filter pad with dilute ammonium hydroxide (1 : 1) until all of the tungstic acid has been dissolved and collected. Add a few ml of the main filtrate (end of 7.4.1) to this solution and slightly acidify with dilute nitric acid (1 : 1). Add dilute ammonium hydroxide (1 : 1) in slight excess and heat to flocculation. Filter through Whatman filter paper No. 42 and wash with dilute ammonium hydroxide (1 : 20). Dissolve the precipitate in minimum hot dilute nitric acid (1 : 5) and add to the main filtrate (7.4.1).

7.4.3 Treat the filtrate as in 6.2 and 6.3. If the sample contains vanadium, proceed further as in 7.3.2. In the absence of vanadium, complete the determination as in 6.4 to 6.6.

7.5 Austenitic Manganese Steels

Decompose 2 g of the sample in a 400 ml beaker in 40 ml of dilute nitric acid (1 : 3). Add 15 ml of perchloric acid and evaporate just to white fumes. Add hydrofluoric acid drop by drop until all the hydrated silica is dissolved and a few drops in excess. Heat so that perchloric acid refluxes on the sides of the beaker for about 25 min. Cool, add about 40 ml of water and 10 ml concentrated nitric acid and boil for few minutes. Proceed further as in 6.2 to 6.6.

7.6 Steels Containing High Arsenic up to 0.1 Percent

Decompose 2 g of the sample in 400 ml beaker in 40 ml of dilute nitric acid (1 : 3). Add 20 ml of perchloric acid and fume to expel nitric acid. Cool, add about 80 ml of dilute hydrobromic acid and evaporate to copious fumes. Cool, add about 40 ml of water and 10 ml nitric acid. Proceed further as in 6.2 to 6.6.

8 BLANK DETERMINATION

Make a blank determination following the same procedure and using the same quantity of all reagents.

9 CALCULATION

9.1 Calculate phosphorus, percent, as:

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

where

B = millilitres of standard nitric acid solution required in the blank determination (*see* 8);

A = millilitres of standard nitric acid solution required in the sample in the titration of the excess sodium hydroxide (*see* 6.6);

C = phosphorus equivalent of 1 ml standard nitric acid solution; and

D = quantity of sample, in g.

9.2 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	MS SANJUKTA A. KUMAR SHRI M. V. RANA (*Alternate*)
CSIR - National Metallurgical Laboratory, Jamshedpur	DR ASHOK K. MOHANTY
Defence Metallurgical Research Laboratory, Ministry of Defence, Hyderabad	SHRI S. S. KALYAN KAMAL
Directorate General of Quality Assurance, Ministry of Defence, New Delhi	SHRI A. MITRA SHRI D. KARTIKEY (*Alternate*)
Dr Jawaharlal Nehru Aluminium Research Development and Design Centre, Nagpur	DR UPENDRA SINGH
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
JSW Steel Limited, Mumbai	SHRI KOTRABASAVARAJU SHRI MARULASIDDESHA U. M. (*Alternate*)
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Shriram Institute for Industrial Research, Delhi	DR LAXMI RAWAT SHRI PUNEET KAPOOR (*Alternate*)
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Tata Steel Limited, Kolkata	DR JATIN MOHAPATRA DR RAVIKRISHNA CHATTI (*Alternate*)
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 'C'/DEPUTY DIRECTOR
(METALLURGICAL ENGINEERING), BIS

(*Continued from second cover*)

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*)’. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Bureau of Indian Standards

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Amendments Issued Since Publication

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