IS : 228 (Part 3) - 1987
(Reaffirmed 1997)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 3 DETERMINATION OF PHOSPHORUS BY ALKALIMETRIC METHOD

(Third Revision)

Fourth Reprint NOVEMBER 1998

UDC 669.14+669.15-194:543.241[543.847]

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *September* 1987

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 3 DETERMINATION OF PHOSPHORUS BY ALKALIMETRIC METHOD

(Third Revision)

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, SMDC 2

Chairman	*Representing*
DR C. S. P. IYER	Bhabha Atomic Research Centre, Bombay
Members	
SHRI G. M. APPARAO	Steel Authority of India Ltd (Bhilai Steel Plant), Bhilai
SHRI R. D. AGARWAL (*Alternate*)	
SHRI S. V. BHAGWAT	Khandelwal Ferro Alloys Ltd, Nagpur
SHRI D. N. GUPTA (*Alternate*)	
SHRI P. CHAKRA	Indian Metals & Ferro Alloys Ltd, Koraput
CHEMIST & METALLURGIST	Ministry of Transport (Department of Railways)
ASSISTANT RESEARCH OFFICER (MET-2), RDSO, LUCKNOW (*Alternate*)	
CHIEF CHEMIST	Tata Iron & Steel Co Ltd, Jamshedpur
ASSISTANT CHIEF CHEMIST (*Alternate*)	
SHRI M. K. CHAKRAVARTY	Ministry of Defence (DGI)
SHRI P. K. SEN (*Alternate*)	
DR M. M. CHAKRABORTY	Indian Iron & Steel Co Ltd, Burnpur
SHRI M. S. CHATTERJEE (*Alternate*)	
SHRI C. K. DIKSHIT	Ordnance Factory Board (Ministry of Defence), Calcutta
SHRI S. N. MOITRA (*Alternate*)	
SHRI V. B. KHANNA	Directorate General of Supplies & Disposals, New Delhi
SHRI J. N. MUKHERJEE	Steel Authority of India Ltd (Durgapur Steel Plant), Durgapur

(*Continued on page* 2)

(*Continued from page* 1)

Members	*Representing*
SHRI P. NARAIN	Mahindra Ugine Steel Co Ltd, Bombay
SHRI G. R. SARMA (*Alternate*)	
SHRI N. P. PANDA	Steel Authority of India Ltd (Rourkela Steel Plant), Rourkela
SHRI B. MAHAPATRA (*Alternate*)	
DR L. P. PANDEY	National Metallurgical Laboratory (CSIR), Jamshedpur
DR D. C. PRASHAR	National Physical Laboratory (CSIR), New Delhi
SHRI J. RAI (*Alternate*)	
SHRI G. RAJARAO	Ferro Alloys Corporation Ltd, Shreeramnagar
SHRI K. RAMAKRISHNAN	Essen & Co, Bangalore
DR J. RAJARAM (*Alternate*)	
SHRI A. P. SINHA	Steel Authority of India Ltd (Bokaro Steel Plant), Bokaro
SHRI K. ANNIAH (*Alternate*)	
SHRI N. V. SUBBARAYAPPA	Visvesvaraya Iron & Steel Ltd, Bhadravati
DR P. SUBRAHMANIAM	Defence Metallurgical Research Laboratory, Hyderabad
SHRI T. H. RAO (*Alternate*)	
DR CH. VENKATESWARLU	Bhabha Atomic Research Centre, Bombay
SHRI K. RAGHAVENDRAN, Director (Struc & Met)	Director General, BIS (*Ex-officio Member*)

Secretary

SHRI M. L. SHARMA
Assistant Director (Metals), BIS

Ferrous Metals Analysis Subcommittee, SMDC 2 : 3

Convener	
DR C. S. P. IYER	Bhabha Atomic Research Centre, Bombay
Members	
SHRI S. BASKARAN	Bharat Heavy Electricals Ltd, Hyderabad
SHRI MATA SARAN (*Alternate* I)	
SHRI B. RAHA (*Alternate* II)	
SHRI U. P. BOSE	Steel Authority of India Ltd (Bhilai Steel Plant), Bhilai
SHRI E. M. VERGHESE (*Alternate*)	
CHIEF CHEMIST	Tata Iron & Steel Co Ltd, Jamshedpur
ASSISTANT CHIEF CHEMIST (*Alternate*)	
DR M. M. CHAKRABORTY	Indian Iron & Steel Co Ltd, Burnpur
SHRI L. N. DAS (*Alternate*)	
SHRI H. K. DAS	Steel Authority of India Ltd (Rourkela Steel Plant), Rourkela
SHRI K. BISHNOI (*Alternate*)	
SHRI A. K. GUPTA	National Physical Laboratory (CSIR), New Delhi

(*Continued on page* 11)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 3 DETERMINATION OF PHOSPHORUS BY ALKALIMETRIC METHOD

(Third Revision)

0. FOREWORD

0.1 This Indian Standard (Part 3) (Third Revision) was adopted by the Indian Standards Institution on 16 January 1987, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 228, which was issued as a tentative standard in 1952 and revised in 1959, covered the chemical analysis of pig iron, cast iron and plain carbon and low alloy steels. For the convenience it was decided to publish a comprehensive series on chemical analysis of steels including high alloy steels. Accordingly, revision of IS : 228 was taken up again and new series on methods of chemical analysis of steels including high alloy steels was published in various parts as IS : 228 (Parts 1 to 13) (*see* Appendix A) covering separate method of analysis for each constituent in steels. However, IS : 228-1959* version has been retained for the analysis of pig iron and cast iron till a separate standard for analysis of pig iron and cast iron is published.

0.2.1 This revision of IS : 228 (Part 3)-1972† has been undertaken on the basis of experience gained during the implementation of the standard by the manufacturers and testing laboratories.

0.3 In this revision method for determination of phosphorus in following alloy steels has been incorporated:

a) Stainless steel, high chromium, nickel chromium and similar alloy steels without tungsten or vanadium,

*Methods of chemical analysis of pig iron, cast iron and plain carbon and low alloy steels (*revised*).

†Methods for chemical analysis of steels: Part 3 Determination of phosphorus by alkalimetric method (*second revision*).

b) Steel containing high silicon, titanium or zirconium,

c) Chromium vanadium steels or other steels containing vanadium but no tungsten,

d) High speed steels or other steels containing tungsten with or without vanadium,

e) Austenitic manganese steels, and

f) Steels containing arsenic up to 0·1 percent.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

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

2. SAMPLING

2.1 The samples shall be drawn and prepared as prescribed in the relevant Indian Standard.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water (*see* IS : 1070-1977†) shall be employed in the test.

4. OUTLINE OF THE METHOD

4.1 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.

4.2 Reagents

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

4.2.2 *Potassium Permanganate Solution* — Dissolve 2 g of potassium permanganate in 100 ml of water.

*Rules for rounding off numerical values (*revised*).

†Specification for water for general laboratory use (*second revision*).

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

4.2.4 *Concentrated Ammonium Hydroxide* — Relative density 0·90.

4.2.5 *Concentrated Nitric Acid* — Relative density 1·42 (conforming to IS : 264-1976*).

4.2.6 *Ammonium Molybdate Solution* — Add solution A (*see* **4.2.6.1**) slowly and with constant stirring to solution B (*see* **4.2.6.2**) 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 Whatman filter paper No. O40 before use.

4.2.6.1 *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.

4.2.6.2 *Solution B* — Add 490 ml of concentrated nitric acid to 1 150 ml of water and cool.

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

4.2.8 *Phenolphthalein Solution* — 1 percent. Dissolve 1 g of powder phenolphthalein in 100 ml of rectified spirit or methyl alcohol.

4.2.9 *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 (**4.2.10**).

4.2.10 *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 ignited at 350°C and cooled.

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

4.2.12 *Hydrofluoric Acid* — 40 percent.

4.2.13 *Perchloric Acid* — 70 percent.

4.2.14 *Sodium Carbonate* — 5 percent (*m/v*).

4.2.15 *Ferrous Sulphate Solution* — Dissolve 100 g of ferrous sulphate crystals ($FeSO_4$, $7H_2O$) in one litre of dilute sulphuric acid (5 : 95).

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

4.2.17 *Sulphurous Acid* — Saturate water with sulphur dioxide gas.

*Specification for nitric acid (*second revision*).

4.2.18 *Concentrated Hydrochloric Acid* — Relative density 1·16 (conforming to IS : 265-1976*).

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

5. PROCEDURE FOR PLAIN CARBON STEEL AND LOW ALLOY STEEL WITHOUT VANADIUM

5.1 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.

5.2 Add 5 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.

5.3 Add ammonium hydroxide till brown precipitate of ferric hydroxide appears. Dissolve the precipitate adding dilute nitric acid (1 : 1) dropwise and add 2 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 5-10 percent.

5.4 Warm the solution to about 60-80°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.

5.5 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).

5.6 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 1-2 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.

*Specification for hydrochloric acid (*second revision*).

6. PROCEDURE FOR ALLOY STEELS

6.1 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 **5.2** to **5.6**.

6.2 Steel Containing High Silicon, Titanium or Zirconium

6.2.1 Follow the procedure given in **6.1** up to filtration and proceed as follows.

6.2.2 Collect the filtrate in a 500-ml conical flask and preserve it. Take the precipitate and pulp (as in **6.1**) 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 **5.2** to **5.6**.

6.3 Chromium Vanadium Steels or Other Steels Containing Vanadium But No Tungsten

6.3.1 Proceed as in **6.1** when the solution is ready for the precipitation of phosphomolybdate complex.

6.3.2 Cool the solution to 10-20°C, 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 **5.5** to **5.6**.

6.4 High Speed Steels or Other Steels Containing Tungsten with or Without Vanadium

6.4.1 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.

6.4.2 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 **6.4.1**) 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 (*see* **6.4.1**).

6.4.3 Treat the filtrate as in **5.2** and **5.3**. If the sample contains vanadium, proceed further as in **6.3.2**. In the absence of vanadium, complete the determination as in **5.4** to **5.6**.

6.5 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 minutes. Cool, add about 40 ml of water and 10 ml concentrated nitric acid and boil for few minutes. Proceed further as in **5.2** to **5.6**.

6.6 Steels Containing High Arsenic Up to 0·1 Percent

6.6.1 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 expell 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 **5.2** to **5.6**.

7. BLANK DETERMINATION

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

8. CALCULATION

8.1 Calculate phosphorus, percent, as:

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

where

A = millilitres of standard nitric acid solution required in the sample in the titration of the excess sodium hydroxide (*see* **5.6**),

B = millilitres of standard nitric acid solution required in the blank determination (*see* **7.1**),

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

D = quantity of sample in g.

8.2 Reproducibility — ± 0·001 5 percent.

APPENDIX A

(*Clause* 0.2)

IS : 228 Methods for chemical analysis of steels:

(Part 1)-1972 Determination of carbon by volumetric method (for carbon ≥ 0·1 percent) (*second revision*)

(Part 2)-1972 Determination of manganese in plain carbon and low alloy steels by arsenite method (*second revision*)

(Part 3)-1972 Determination of phosphorus by alkalimetric method (*second revision*)

(Part 4)-1974 Determination of carbon by gravimetric method (for carbon ≥ 0·1 percent) (*second revision*)

(Part 5)-1974 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel ≥ 0·5 percent) (*second revision*)

(Part 6)-1974 Determination of chromium by persulphate oxidation method (for chromium ≥ 0·5 percent) (*second revision*)

(Part 7)-1974 Determination of molybdenum by α-benzoinoxime method (for molybdenum > 1 percent) (*second revision*)

(Part 8)-1975 Determination of silicon by the gravimetric method (for silicon ⩾ 0·1 percent) (*second revision*)

(Part 9)-1975 Determination of sulphur in plain carbon steels by evolution method (*second revision*)

(Part 10)-1976 Determination of molybdenum by thiocyanate (photometric) method (for molybdenum up to 1 percent) in low and high alloy steels (*second revision*)

(Part 11)-1976 Determination of silicon by photometric method in carbon steels and low alloy steels (for silicon 0·01 to 0·05 percent) (*second revision*)

(Part 12)-1976 Determination of manganese by periodate (photometric) method in low and high alloy steels (for manganese up to 2 percent) (*second revision*)

(Part 13)-1982 Determination of arsenic

(*Continued from page* 2)

Members	*Representing*
SHRI J. MUKHERJEE	Steel Authority of India Ltd (Durgapur Steel Plant), Durgapur
SHRI P. K. BANERJEE (*Alternate*)	
SHRI P. NARAIN	Mahindra Ugine Steel Co Ltd, Bombay
SHRI G. R. SARMA (*Alternate*)	
SHRI R. S. NATH	Steel Authority of India Ltd (Bokaro Steel Plant), Bokaro
SHRI N. GUNDAPPA (*Alternate*)	
DR L. P. PANDEY	National Metallurgical Laboratory (CSIR), Jamshedpur
SHRI G. RAMDAS	Visvesvaraya Iron & Steel Ltd, Bhadravati
SHRI R. D. VANDRIWALLA	Italab Pvt Ltd, Bombay
SHRI J. C. DEY (*Alternate*)	

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones: 323 0131, 323 3375, 323 9402
Fax : 91 11 3234062, 91 11 3239399, 91 11 3239382

Telegrams : Manaksanstha
(Common to all Offices)

Central Laboratory:	Telephone
Plot No. 20/9, Site IV, Sahibabad Industrial Area, SAHIBABAD 201010	8-77 00 32
Regional Offices:	
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	323 76 17
*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	337 86 62
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9 Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	832 92 95
Branch Offices:	
'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	550 13 48
‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar, BHOPAL 462003	55 40 21
Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28 88 01
Savitri Complex, 116 G. T. Road, GHAZIABAD 201001	8-71 19 96
53/5 Ward No. 29, R. G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-58C, L. N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	20 10 83
E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001	37 29 25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
Patliputra Industrial Estate, PATNA 800013	26 23 05
T. C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034	6 21 17
NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005	32 36 35

*Sales Office is at 5 Chowringhee Approach, P. O. Princep Street, CALCUTTA 700072 — 27 10 85

†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007 — 309 65 28

‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002 — 222 39 71

Printed at New India Printing Press, Khurja, India