भारतीय मानक
Indian Standard

IS 228 (Part 6) : 2024

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 6 पर्सल्फेट ऑक्सीकरण पद्धति द्वारा क्रोमियम का निर्धारण (क्रोमियम ≥ 0.1 प्रतिशत के लिए)

(चौथा पुनरीक्षण)

Methods for Chemical Analysis of Steels

Part 6 Determination of Chromium by Persulphate Oxidation Method (for Chromium ≥ 0.1 Percent)

(Fourth Revision)

ICS 77.080.20

भारतीय मानक ब्यूरो
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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 6) (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, 1974 and 1987, covered the chemical analysis of plain carbon and low alloy steels, along with pig iron and cast iron. 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 chromium by persulphate oxidation 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 3	Determination of phosphorus by alkalimetric method
Part 4	Determination of total carbon by gravimetric method (for carbon ≥ 0.1 percent)
Part 5	Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel ≥ 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 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 1.50 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 0.001 to 0.100 0 percent)
Part 19	Determination of nitrogen by steam distillation method (for nitrogen 0.002 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 0.50 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)

(*Continued on third cover*)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 6 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION METHOD (FOR CHROMIUM ≥ 0.1 PERCENT)

(Fourth Revision)

1 SCOPE

This standard (Part 6) covers the persulphate oxidation method for determination of chromium content of low alloy and high alloy steels containing more than or equal to 0.1 percent chromium. This method is not applicable for steels containing tungsten.

2 REFERENCES

The following 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 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 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION METHOD

5.1 Outline of the Method

After dissolution of the sample in dilute sulphuric acid and phosphoric acid mixture and further treated with nitric acid; chromium, manganese and (vanadium if present) are oxidized by ammonium persulphate in presence of silver nitrate as catalyst. Permanganic acid is then destroyed by dilute hydrochloric acid. Chromium is reduced by ferrous ammonium sulphate and excess of ferrous ammonium sulphate is back titrated with standard potassium permanganate solution.

5.2 Reagents

5.2.1 *Phosphoric Acid - Sulphuric Acid Mixture*

To 600 ml of water, add continuously 165 ml of concentrated sulphuric acid (rd = 1.84) and 132 ml of phosphoric acid (rd = 1.75). Mix, cool and dilute to 1 litre.

5.2.2 *Concentrated Nitric Acid*

Relative density = 1.42 (conforming to IS 264).

5.2.3 *Silver Nitrate Solution* — 0.5 percent (*m/v*)

Dissolve 5 g of silver nitrate crystals in water and dilute to 1 litre.

5.2.4 *Ammonium Persulphate Solution*

Dissolve 15 g of ammonium persulphate in 100 ml of water. Use a freshly prepared solution.

5.2.5 *Potassium Permanganate Solution* — 1 percent (*m/v*)

5.2.6 *Dilute Hydrochloric Acid* — 1 : 3 (*v/v*)

Dilute 250 ml of concentrated hydrochloric acid (rd = 1.16) to 1 litre.

5.2.7 *Standard Ferrous Ammonium Sulphate Solution* — approximately 0.1 N

Dissolve 40 g of ferrous ammonium sulphate in sulphuric acid (5 percent) and dilute to 1 litre. Filter, if necessary, and keep in a stoppered glass bottle. Standardize against standard potassium permanganate solution (given under **5.2.8**) every time it is used.

5.2.8 *Standard Potassium Permanganate Solution* — approximately 0.1 N

Dissolve 3.2 g of potassium permanganate crystals in 1 000 ml of water, stir and allow to stand in a closed vessel for 24 h. Filter, through a sintered glass crucible and keep in an amber-coloured glass bottle. Standardize the solution as follows:

Dissolve 0.134 g of sodium oxalate crystals, dried for 1 hour at 105 °C in 200 ml of dilute sulphuric acid (1 : 50). Heat to 70 °C and titrate with potassium permanganate solution until one drop produces a permanent pink colouration. [1 ml of potassium permanganate solution (0.1 N) = 0.006 7 g of sodium oxalate].

5.3 Procedure

5.3.1 Take 2 g of sample (for chromium less than 2 percent) and 0.2 g to 0.5 g of sample for high alloy steels in a wide mouth conical flask. Add 50 ml of phosphoric acid-sulphuric acid mixture. Heat the flask to decompose the sample. Oxidize black residue by addition of concentrated nitric acid dropwise and heating the solution simultaneously till all carbides are decomposed and brown fumes are expelled. Dilute to 300 ml with hot water.

5.3.2 Add a few pieces of glass beads, heat the solution to boiling and add 20 ml of silver nitrate solution and 20 ml of ammonium persulphate solution adding little at a time and continue boiling till the permanganate colour develops fully (volume should be maintained at 300 ml by addition of hot water, if necessary and also boiling should be a period of 8 min to 10 min). It should be ensured that sufficient persulphate is added. Wash the sides of the conical flask with water. If the colour does not develop add a few drops of potassium permanganate solution till the pink colour develops.

5.3.3 Add dilute hydrochloric acid dropwise to the boiling solution till permanganic acid colour is destroyed. Boil for 10 min more. Cool and add a known volume of standard ferrous ammonium sulphate solution until an excess of at least 5 ml is present. Titrate back with dropwise addition of standard potassium permanganate solution to a permanent pink end point which persists for 30 s to 40 s.

5.3.4 In presence of vanadium, titrate carefully to a pink end point which persists for at least. 30 s to 40 s, to ensure complete re-oxidation of the vanadium.

5.3.5 The titration should be corrected for dilution effect and colour interference. The correction may be made by the following method:

Add same amount of ferrous ammonium sulphate as used for the sample, to the already titrated solution. Titrate with standard potassium permanganate to pink end point which lasts for 30 s to 40 s.

5.4 Calculation

5.4.1 Calculate the chromium content of the steel as follows:

$$\text{Chromium, percent} = \frac{(AB - C)\, D \times 0.017\ 33 \times 100}{E}$$

where

A = volume, in ml, of standard ferrous ammonium sulphate solution added;

B = volume, in ml, of standard potassium permanganate solution equivalent to 1 ml of ferrous ammonium sulphate solution;

C = volume, in ml, of standard potassium permanganate solution required for titration, corrected for the blank;

D = normality of standard potassium permanganate solution; and

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

5.4.2 *Reproducibility*

a) ± 0.025 percent at 0.1 to 0.5 percent chromium;

b) ± 0.036 percent at 0.5 to 1 percent chromium;

c) ± 0.l20 percent at 1 to 5 percent chromium; and

d) ± 0.20 percent for chromium 5 percent and above.

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 (*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 A. MITRA SHRI D. KARTIKEY (*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
JSW Steel Limited, Mumbai	SHRI KOTRABASAVARAJU SHRI MARULASIDDESHA U. M. (*Alternate*)
Jawaharlal Nehru Aluminium Research Development and Design Centre, Nagpur	DR UPENDRA SINGH
National Aluminium Company Limited, Bhubaneswar	SHRIMATI SUKLA NANDI SHRI DEBANANDA BHATTACHARYYA (*Alternate*)
National Test House, Kolkata	DR RAJEEV KUMAR UPADHYAY SHRI AKBAR H. (*Alternate*)
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*)
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*)

Part 24 Determination of nitrogen in steel by inert gas fusion — Thermal conductivity method (nitrogen 0.001 to 0.2 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*)’. 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 are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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