भारतीय मानक

Indian Standard

IS 228 (Part 4) : 2024

इस्पात के रासायनिक विश्लेषण की पद्धति

भाग 4 ग्रेविमेट्रिक विधि द्वारा कुल कार्बन का निर्धारण (कार्बन ≥ 0.1 प्रतिशत के लिए)

(चौथा पुनरीक्षण)

Method for Chemical Analysis of Steels

Part 4 Determination of Total Carbon by Gravimetric Method (for Carbon ≥ 0.1 Percent)

(Fourth Revision)

ICS 77.080.20

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 (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. It was revised again to make it comprehensive in respect of steel analysis and to exclude pig iron and cast iron which were being covered in separate standards. During its second revision the standard has been split up in several parts.

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 determination of total carbon content of plain carbon, low alloy and high alloy steels of 0.1 percent and above by the gravimetric 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 5	Determination of nickel by dimethyl glyoxime (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 (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 1.5 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 0.002 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 0.5 percent)
Part 22	Determination of total hydrogen in steel by thermal conductivity method (hydrogen 0.1 ppm to 50 ppm)

(*Continued on third cover*)

Indian Standard

METHOD FOR CHEMICAL ANALYSIS OF STEELS

PART 4 DETERMINATION OF TOTAL CARBON BY GRAVIMETRIC METHOD (FOR CARBON ≥ 0.1 PERCENT)

(Fourth Revision)

1 SCOPE

This standard (Part 4) covers the method for determination of total carbon content of plain carbon, low alloy and high alloy steels of 0.1 percent and above by the gravimetric method.

2 REFERENCES

This standard given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated was valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of this standard:

IS No	*Title*
IS 6226 (Part 1) : 1994	Recommendations of apparatus for chemicals analysis of metals: Part 1 Apparatus for determination of carbon by direct combustion (*first revision*)

3 OUTLINE OF THE METHOD

The sample is burnt in a stream of purified oxygen and the carbon dioxide formed is absorbed, after purification, in suitable absorbent and determined.

4 REAGENTS

4.1 Oxygen (O_2) — 99.5 percent minimum

4.2 Accarite or Soda Lime — 0.80 mm to 2.00 mm

4.3 Magnesium Perchlorate — Mg $(ClO_4)_2$, 0.80 mm to 2.00 mm

4.4 Boat/Crucible

Boat/crucible of precise dimension for accommodating in the resistance and induction furnace.

Pre-ignite the boats/crucibles in air or oxygen in a furnace for an hour at 1 100 °C and store in a desiccator and check for consistency of the blank values.

4.5 Flux/Accelerator — low carbon copper, red lead (pre-ignited at 550 ºC), tin and iron of low carbon content.

5 APPARATUS

5.1 The apparatus recommended in IS 6226 (Part l) may be used.

5.2 Instead of the resistance furnace, an induction furnace may also be used.

6 SAMPLING

6.1 The sample shall be drawn as prescribed in the relevant Indian Standards.

6.2 The sample is to be cleaned with analytical grade ether and acetone, dried in an air oven at 100 °C ± 5 °C.

7 PROCEDURE

7.1 Assemble the apparatus. Switch on the furnace, if it is a resistance furnace, and allow it to attain a temperature of 1 050 ºC (*see* Note), all the while passing oxygen through the apparatus so that it bubbles freely at the exit end of the train. Disconnect the absorption bulb, keep in a desiccator till it attains room temperature and take the initial weight. Repeat the operation till a constant weight is obtained.

NOTE — For high chromium and high nickel steel, the temperature of 1 250 °C is recommended for complete combustion.

7.2 Weigh to the nearest 0.001 g, 2.0 g to 3.0 g of the test sample. Transfer to the pre-ignited combustion boat covered at the bottom with a thin layer of calcined alumina. Spread the sample evenly over the top of the alumina and cover it with 2.0 g to 3.0 g of the flux. Introduce the boat slowly in the hot zone of the combustion tube.

7.3 In the case of induction heating, weigh to the nearest 0.001 g, 0.9 g to 1.1 g of the sample and transfer to a pre-ignited crucible. Add an equal quantity of the flux. Place the crucible in position on the pedestal post of the furnace, raise to the combustion position and lock the system. Pass oxygen through the system and ignite the sample.

7.4 Maintain a rapid flow of oxygen (800 ml/min to 1 000 ml/min) throughout the combustion, then reduce to 400 ml to 500 ml per minute and maintain it for another 6 min to 8 min in order to sweep out the carbon dioxide.

7.5 Remove the absorption bulb and weigh it after keeping it in desiccator till it attains room temperature. The increase in weight of the bulb represents the carbon dioxide.

7.6 Remove the boat or crucible and examine for any incomplete combustion. If the sample is not thoroughly fused, repeat the determination with a fresh sample.

7.7 Blank

Charge a pre-ignited boat or crucible, as the case may be, with the same amount of flux used in the determination and follow the procedure as in 7.2 to 7.5.

8 CALCULATION

8.1 Calculate the total carbon content of the sample as follows:

$$\text{Carbon, percent} = \frac{A - B}{C} \times 27.29$$

where

A = increase in mass, in g, of the absorption bulb due to carbon dioxide from the sample;

B = increase in mass, in g, of the absorption bulb due to carbon dioxide from the blank determination; and

C = mass, in g, of the sample taken.

9 ACCURACY

The accuracy of the method is ± 0.01 percent for carbon the range of 0.1 percent to 0.75 percent and ± 0.02 percent for carbon above 0.75 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 (*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
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 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	SHRI 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 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)

The composition of the Committee responsible for 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

Amend No.	Date of Issue	Text Affected

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