For Comments Only

BUREAU OF INDIAN STANDARDS

***Draft* Revision**

TO

IS 228 (Part 4): METHODS FOR CHEMICAL ANALYSIS OF STEELS

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

(Fourth Revision)

Last date for receipt of comments is XXXX

भारतीय मानक प्रारूप

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 4 XXXX

(चौथा पुनरीक्षण)

Draft Indian Standard

METHODS 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

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

XXXX **2023** **Price Group**

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This draft Indian Standard (Part 4) (Fourth Revision) subject to its finalization, is to be adopted by the Bureau of Indian Standards on recommendation of the Methods of Chemical analysis of Metals Sectional Committee and approval of 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 greater than or equal to 0.1 percent)
Part 6	Determination of chromium by persulphate oxidation method (for chromium $\geq$ 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 in plain carbon steels 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 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

Part 18 Determination of oxygen by instrumental method

Part 19 Determination of nitrogen by steam distillation

Part 20 Determination of carbon and sulphur by infrared absorption 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)

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.

Draft Indian Standard

METHODS 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

The following Indian Standards 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 editions of these standards indicated below:

IS No	*Title*
IS 6226 (Part 1) : 1994	Recommendations for 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 to 2.00 mm.

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

4.4 Boat/Crucible

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

4.4.1 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 analar grade ether and acetone, dried in an air oven at 100 ± 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 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 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 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 to 1 000 ml/min) throughout the combustion, then reduce to 400 to 500 ml per min and maintain it for another 6 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 to 0.75 percent and ± 0.02 percent for carbon above 0.75 percent.