Indian Standard

METHOD FOR CHEMICAL ANALYSIS OF STEELS

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

(Third Revision)

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BUREAU OF INDIAN STANDARDS
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PART 4 DETERMINATION OF TOTAL CARBON BY GRAVIMETRIC METHOD (FOR CARBON > 0·1 PERCENT)

(*Third Revision*)

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(*Continued on page* 2)

(*Continued from page* 1)

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(*Continued on page* 8)

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METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 4 DETERMINATION OF TOTAL CARBON BY GARVIMETRIC METHOD (FOR CARBON ≥ 0·1 PERCENT)

(Third Revision)

0. FOREWORD

0.1 This Indian Standard (Part 4) (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 method 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 4)-1974† 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 has been updated.

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

†Methods for chemical analysis of steels: Part 4 Determination of carbon by gravimetric method (for carbon ≥ 0·1 percent) (*second revision*).

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 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. OUTLINE OF THE METHOD

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

3. REAGENTS

3.1 Oxygen (O_2) — 99·5 percent minimum.

3.2 Accarite or Soda Lime – 0·80 to 2·00 mm.

3.3 Magnesium Perchlorate — $Mg(ClO_4)_2$, 0·80 to 2·00 mm.

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

3.4.1 Preignite 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 consistancy of the blank values.

3.5 Flux/Accelerator — Low carbon copper, red lead (preignited at 550°C), tin and iron of low carbon content.

4. APPARATUS

4.1 The apparatus recommended in IS : 6226 (Part 1)-1971† may be used.

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

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

†Recommendation for apparatus for chemical analysis of metals: Part 1 Determination of carbon by direct combustion.

5. SAMPLING

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

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

6. PROCEDURE

6.1 Assemble the apparatus. Switch on the furnace, if it is a resistance furnace, and allow it to attain a temperature of 1 050°C (*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 steels, the temperature of 1 250°C is recommended for complete combustion.

6.2 Weigh to the nearest 0·001 g, 2·0 to 3·0 g of the test sample. Transfer to the preignited 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.

6.3 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 preignited 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.

6.4 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.

6.5 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.

6.6 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.

6.7 Blank — Charge a preignited boat or crucible, as the case may be, with the same amount of flux used in the determination and follow the procedure as in **6.2** to **6.5**.

7. CALCULATION

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

$$\text{Carbon, percent} = \frac{A - B}{C} \times 27{\cdot}29$$

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.

8. ACCURACY

8.1 The accuracy of the method is $\pm$ 0·01 percent for carbon in the range of 0·1 to 0·75 percent and $\pm$ 0·02 percent for carbon above 0·75 percent.

APPENDIX A

(*Clause* 0.2)

INDIAN STANDARDS ON METHODS FOR CHEMICAL ANALYSIS OF STEELS

IS : 228 Methods for chemical analysis of steels:

(Part 1)-1972 Determination of carbon by volumetric method (for carbon $\geq$ 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)-1987 Determination of total carbon by gravimetric method (for carbon $\geq$ 0·1 percent) (*third revision*)

(Part 5)-1974 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel $\geq$ 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

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