Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 14 DETERMINATION OF CARBON BY THERMAL CONDUCTIVITY METHOD (FOR CARBON 0.005 TO 2.000 PERCENT)

1. Scope — This standard (Part 14) covers a method for determination of carbon in all types of steels and alloy steels in the range of 0'005 to 2'000 percent.

2. Determination of Carbon by Thermal Conductivity Method

2.1 Outline of the Method — The sample is burnt in a stream of oxygen in presence of a metal accelerator. The carbon dioxide formed is selectively adsorbed on the molecular sieve at a temperature and released by heating at 300°C. The detector is a thermistor cell which senses the difference between thermal conductivity of the carrier gas (with helium specially for extra-low carbon, and oxygen in other cases) and that of the carrier gas containing carbon dioxide. This difference is proportional to carbon content of the sample.

3. Reagents

- 3.1 Oxygen (O₂) 99 5 percent pure, Min.
- 3.2 Helium 99'5 percent pure, Min.

3.3 Ascarite or Soda-Lime - 0'80 mm-2'0 mm.

3.4 Magnesium Perchlorate - 0'80 mm-2'0 mm.

3.5 Concentrated Sulphuric Acid (rd = 1.84) — Conforming to IS: 263-1977 'Specification for sulphuric acid (second revision)'.

3.6 Sulphur Trap — containing manganese dioxide (MnO₂).

3.7 Carbon Dioxide Converter - containing copper oxide maintained at 300°C.

3.8 Accelerators -- copper, tin or iron granules, free from carbon and sulphur.

3.9 Crucibles — pre-ignited crucibles of precise dimensions which may be accommodated in combustion tube of the induction furnace.

4. Apparatus — Any analyser consisting of induction furnace, molecular sieve, chromatographic column and thermistor type detector.

5. Sampling — The samples shall be drawn and prepared as prescribed in the relevant Indian Standard.

6. Procedure

6.1 Standardization

6.1.1 Switch on the instrument for 4 hours before analyzing the samples for attaining thermal stability of the cell.

6.1.2 Start the flow of purified oxygen gas and pass it continuously through the system at the rate of $1\ 000\ -1\ 500\ ml/minute$.

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6.1.3 Transfer into the pre-ignited crucible 1.00 g standard sample which has a value of carbon in the range of interest and add 1.0 g accelerator.

6.1.4 Insert the crucible into the induction furnace, wait for 30 seconds and start the induction.

6.1.5 Note the percentage carbon, and adjust if necessary, the standardization until the certified value of carbon for the standard sample is obtained and with the desired reproducibility.

6.2 For Sample

6.2.1 Transfer 1 g of accurately weighed sample previously washed with organic solvent (like acetone, benzene or ether) thrice and dried at $100\pm5^{\circ}$ C on the crucible and add 1.0 g of accelerator.

6.2.2 Insert into the induction furnace and proceed until the percentage of carbon is read out.

7. Reproducibility – \pm 0.000 2 percent or \pm 0.5 percent of carbon present whichever is greater.

EXPLANATORY NOTE

The first revision of IS: 228-1959 covered the chemical analysis of plain carbon and low alloy steels along with pig iron and cast iron. This standard was again revised to make it comprehensive in respect of steel analysis and to exclude pig iron and cast iron analysis which is being covered in a separate standard. The second revision of IS: 228 was issued in parts covering chemical analysis of steels. This part (Part 14) covers chemical analysis of carbon in steels by thermal conductivity method. Determination of carbon in steels by infra-red combustion method is being covered in another part of series of this standard. However, determination of carbon in steels by volumetric and gravimetric methods has been prescribed in Part 1 and Part 4 of this standard. The other parts of this series are :

- (Part 1)-1988 Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent) (third revision)
- (Part 2)-1987 Determination of manganese in plain carbon and low alloy steels by arsenite method (*third revision*)
- (Part 3)-1987 Determination of phosphorus by alkalimetric method (third revision)
- (Part 4)-1987 Determination of carbon by gravimetric method (for carbon > 0.1 percent) (third revision)
- (Part 5)-1987 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel ≥ 0.1 percent) (third revision)
- (Part 6)-1987 Determination of chromium by persulphate oxidation method (for chromium ≥ 0.1 percent) (third 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