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Draft Indian Standard

METHODS OF CHEMICAL ANALYSIS OF FERROSILICON

PART 2 DETERMINATION OF CARBON

[Second Revision of IS 1559 (Part 2)]

ICS 77.100

Methods of Chemical Analysis of Metals
Sectional Committee, MTD 34

Last date of comments
08 September 2023

FOREWORD

(Formal foreword clause will be added later)

This standard was first published in 1961 covering chemical analysis of different ferroalloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten. The first revision of this standard was published in seven parts covering chemical analysis of ferrosilicon and by superseding Section I of IS 1559 : 1961. The committee also decided to rewrite the methods taking into consideration the latest composition specified in IS 1110 : 1990. Though calcium and manganese were not specified in IS 1110 : 1990, the determination of these elements are covered in this standard because these are generally required from the view point of determining interference of analysis in methods for determining other elements in ferrosilicon due to presence of these elements. Moreover the determination of these elements may be required for comprehensive analysis of ferrosilicon.

This standard (Part 2) covers the determination of carbon. The other parts in the series are:

- Part 1 Determination of silicon
- Part 3 Determination of sulphur
- Part 4 Determination of phosphorus
- Part 5 Determination of aluminium
- Part 6 Determination of calcium
- Part 7 Determination of manganese

In this part only direct combustion gravimetric method has been retained and the reference of relevant Indian Standard for the apparatus to be used has been given.

This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

In reporting the results 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 : 2022 'Rules for rounding off numerical values (*second revision*)'.

*Draft Indian Standard***METHODS OF CHEMICAL ANALYSIS OF FERROSILICON****PART 2 DETERMINATION OF CARBON***(Second Revision)***1 SCOPE**

This standard (Part 2) covers the determination of carbon (0.15 to 1.50 percent) in ferrosilicon.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were 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 editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
IS 266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 1472 : 1977	Methods of sampling ferro-alloys for determination of chemical composition (<i>first revision</i>)
IS 6226 (Part 1) : 1994	Recommendations for apparatus for chemicals analysis of metals: Part 1 Apparatus for determination of carbon by direct combustion (<i>first revision</i>)

3 SAMPLING

It shall be drawn and prepared in accordance with IS 1472.

4 QUALITY OF REAGENTS

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be employed in the tests.

NOTE – ‘Pure-chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

5 DETERMINATION OF CARBON (TOTAL) BY THE DIRECT COMBUSTION GRAVIMETRIC METHOD**5.1 Outline of the Method**

The sample is burnt in a stream of pure oxygen, and the resulting carbon dioxide is absorbed in soda asbestos after removal of the contaminants. From the increase in weight of the absorbent the carbon content is calculated.

5.2 Apparatus

The apparatus recommended in IS 6226 (Part 1) may be used.

5.3 Reagents

5.3.1 Concentrated Sulphuric Acid — relative density = 1.84 (conforming to IS 266).

5.3.2 Dilute Sulphuric Acid — 2.98 (v/v).

5.3.3 Chromic-Sulphuric Acid Solution — saturate 100 ml of dilute sulphuric acid (2 : 1) with chromic anhydride.

5.3.4 Manganese Sulphate — solid.

5.3.5 Concentrated Ammonium Hydroxide — relative density = 0.90.

5.3.6 Ammonium Persulphate Solution — 25 percent (m/v).

5.3.7 Precipitated Manganese Dioxide

- a) Dissolve 200 g of manganese sulphate ($\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$) in 2 500 ml of water, add concentrated ammonium hydroxide until ammoniacal, followed by 1 000 ml of freshly prepared ammonium persulphate solution (22.5 percent) and heat to boiling. Boil for 10 minutes, adding more of concentrated ammonium hydroxide to maintain the ammoniacal condition.
- b) Discontinue boiling and allow the precipitate to settle; wash by decantation 6 to 8 times with 500 ml to 600 ml portions of water, allowing the precipitate to settle completely between washings. Finally, wash 2 to 3 times by decantation with 500 ml to 600 ml portions of dilute sulphuric acid. Transfer the precipitate to a Buchner funnel and wash with hot water until free from sulphates. Dry the residue at 105 °C to 110 °C, grind to pass IS Sieves 85 to 70 (aperture 842 to 708 microns) and again dry thoroughly at 105 °C to 110 °C.

5.3.8 Soda Asbestos

It should be graded to IS Sieves 140 to 50 (aperture 1 405 to 500 microns).

5.3.9 Anhydrous or Magnesium Perchlorate $\text{Mg}(\text{ClO}_4)_2$ — the supply bottle should be well stoppered.

5.3.10 Lead Foil, this should be of assay grade and 0.127 mm in thickness.

5.3.11 Magnesite Brick Powder, magnesite brick powder which has passed through IS Sieve 50 (aperture 500 microns) shall be used for spreading in the boat on which the sample is to be placed before combustion. This powder shall be calcined in the open muffle furnace prior to use, in order to eliminate any carbonaceous matter.

5.4 Procedure

5.4.1 Heat the furnace to a temperature between 1 250 °C and 1 350 °C. Before starting the analysis, test the apparatus for any leaks. Pass a slow stream of oxygen for about 15 minutes through the whole train of the apparatus and then weigh the carbon dioxide absorption tube. A counterpoise of

a similar bottle is recommended in a balance for weighing. After taking the first weight connect it back to the apparatus, pass oxygen for 15 minutes and weigh again. If constant mass is obtained, the apparatus is ready for use.

5.4.2 Weigh 2.0 g of the sample mixed with 2.0 g of a low carbon standard steel of known carbon content and place in a previously ignited combustion boat made of either porcelain, refractory, clay, nickel-sheet or alumina and of dimensions about $100 \times 20 \times 15$ mm with wall as thin as possible and cover the sample with lead foil (1.5 g twice folded). Before placing the sample in the boat sprinkle in the bottom of the boat some burnt magnesite brick dust which has been tested by the blank run (*see 5.4.3*). Insert the boat quickly into the hot zone of the furnace, with the rubber stopper replaced at the end of the combustion tube, wait for about a minute so that the boat attains the temperature of the furnace and pass a rapid stream (300 ml to 500 ml per minute) of oxygen for 10 to 15 minutes to sweep out carbon dioxide. At the end of this period, remove the carbon dioxide absorption bulb, cool in a desiccator and weigh quickly against a similar tube used as a counterpoise. The increase in mass represents carbon dioxide.

5.4.3 Carry out a blank determination on 2.0 g of the same low carbon standard steel that was mixed with the sample.

5.5 Calculation

$$\text{Carbon, percent} = \frac{(A - B) \times 27.27}{C}$$

where

A = mass in g of carbon dioxide absorption tube after test,

B = mass in g of carbon dioxide absorption tube in blank run, and

C = mass in g of the sample taken.