IS 228 (Part 11) : 1990
(Reaffirmed 1994)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

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)

(Third Revision)

भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 11 कार्बन इस्पात तथा अल्प मिश्र इस्पात (सिलीकॉन के लिए 0·01 से 0·05 प्रतिशत) में अपचित मॉलीब्डोसिलिकेट स्पेक्ट्रम प्रकाशमापी पद्धति द्वारा कुल सिलीकॉन ज्ञात करना

(तीसरा पुनरीक्षण)

UDC 669·14 + 669·15-194·2 : 543·42 [546·28]

BUREAU OF INDIAN STANDARDS
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September 1990 **Price Group 1**

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 11) (Third Revision) was adopted by the Bureau of Indian Standards on 23 February 1990, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 228, which was first published in 1952 and subsequently revised in 1959, 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 was split up in several parts, and 14 parts have already been published covering only chemical analysis of steels.

This standard IS 228 (Part 11) was published in 1976. In this third revision this part has been brought in line with ISO document ISO/DIS 4829-2 'Steel and iron — Determination of total silicon content — Reduced molybdosilicate spectrophotometric method: Part 2 Silicon content between 0·01 and 0·05 percent' and reproducibility of the method has also been incorporated.

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 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

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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)

(Third Revision)

1 SCOPE

1.1 This standard (Part 11) covers the spectrophotometric method for determination of total silicon in the range of 0·01 to 0·05 percent in carbon steels and low alloy steels.

2 SAMPLING

2.1 Samples shall be drawn and prepared as prescribed in the relevant Indian Standards.

3 QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

4 DETERMINATION OF TOTAL SILICON BY REDUCED MOLYBDOSILICATE SPECTROPHOTOMETRIC METHOD

4.1 Outline of the Method

Silicon is converted to silicic acid and complexed with sodium molybdate which in turn is reduced to molybdenum blue. The absorbance of complex is measured at 810 nm.

4.2 Reagents

4.2.1 *Hydrochloric Acid-Nitric Acid Mixture*

Add 180 ml of concentrated hydrochloric acid (rd = 1·16) and 65 ml of concentrated nitric acid (rd = 1·42) to 500 ml water. Cool and dilute to one litre.

4.2.2 *Sodium Carbonate*

4.2.3 *Dilute Sulphuric Acid*, 1 : 4 and 1 : 20 (*v/v*).

4.2.4 *Potassium Permanganate Solution*, 2 percent (*w/v*).

4.2.5 *Hydrogen Peroxide*

Dilute 200 ml of hydrogen peroxide (30 percent) to one litre with water.

4.2.6 *Sodium Molybdate Solution*

Dissolve 2·5 g of sodium molybdate dihydrate ($Na_2MoO_4 \cdot 2H_2O$) in 50 ml of water and filter through medium texture filter paper. Immediately before use, add 15 ml of concentrated sulphuric acid (rd = 1·84), dilute to 100 ml and mix.

4.2.7 *Oxalic Acid*, 5 percent (*w/v*).

4.2.8 *Ascorbic Acid*, 2 percent (prepare immediately before use).

4.2.9 *Pure Iron*, (silicon free). (silicon less than 2 µg/g).

4.2.10 *Standard Silicon Solution* (1 ml = 1 mg Si). Ignite silica (purity $>$ 99·9% SiO_2) at 150°C. Cool and weigh 2·14 g and transfer to a platinum crucible. The high purity silica shall be freshly calcined and cooled in a desiccator. Mix thoroughly with 16 g of anhydrous sodium carbonate and fuse at 1 050°C for 30 minutes. Extract the fused mass with 100 ml of water in polypropylene or PTFE beaker. Transfer to 1 litre volumetric flask and shake well. Transfer the solution immediately to well-stoppered polytetrafluoroethylene bottle for storage.

4.2.10.1 *Standard silicon solution* — (1 ml = 20 µg Si).

Transfer 10 ml of solution (**4.2.10**) to 500 ml volumetric flask. Dilute to the mark and mix. Transfer the solution to polytetrafluoroethylene bottle for immediate use. This solution contains 20 µg of Si per ml.

NOTE — All reagents shall be freshly prepared and stored in polypropylene or polytetrafluoroethylene containers.

4.3 Procedure

4.3.1 Weigh 0·500 g of sample in a 250-ml polypropylene or polytetrafluoroethylene beaker. Add 85 ml of hydrochloric acid-nitric acid mixture, cover with a lid and warm gently to dissolve. When the reaction ceases, filter the solution through close texture ashless filter paper and collect the filtrate. Rinse the beaker with 20 ml water and wash the filter paper several times with 20 ml of hot water. Reserve the filtrate.

4.3.2 Transfer the residue and filter paper to a platinum crucible and ignite at low temperature, until carbonaceous matter is removed. Ignite at 600°C. Cool and mix the residue with 1 g of sodium carbonate and heat at 900°C for 10 minutes. Cool and add 15 ml of water. Cover the crucible and allow the reaction to subside. Add 15 ml of dilute sulphuric acid (1 : 20), stir to dissolve the melt and add to the filtrate (**4.3.1**).

4.3.3 Transfer the solution (**4.3.2**) to a 1-litre volumetric flask and make up with water. Take 20 ml aliquot of the above solution into 50 ml volumetric flask. Add potassium permanganate solution dropwise till pink colour is obtained, which persists for 1 minute. Heat to boiling and boil for 2 minutes. If the percipitate of manganese dioxide occurs, add hydrogen peroxide dropwise till the precipitate is just dissolved and boil for 5 minutes. Add 10 ml of sodium molybdate and allow to stand for 20 minutes. Add 5 ml of dilute sulphuric acid (1 : 4), 5 ml of oxalic acid and 5 ml of ascorbic acid solution.

NOTE — The temperature of the solution be maintained between range 15-25°C.

Dilute to mark and mix. Measure the absorbance of the test solution against compensating solution at 810 nm.

4.3.4 *Compensating Solution*

Take 20 ml aliquot of the sample solution (**4.3.3**) in 50 ml volumetric flask and add the following reagents, shaking after each addition:

- 5 ml of sulphuric acid (1 : 4),
- 5 ml of oxalic acid,
- 10 ml of sodium molybdate and immediately add,
- 5 ml of ascorbic acid.

Dilute to mark and mix well.

4.3.5 *Blank Solution*

Simultaneously with test solution and compensating solution, carry out two blank tests using 0·500 g of pure iron instead of sample.

NOTE — It is recommended that duplicate blanks be run with each of the tests. Find the absorbance values against the compensating solution.

4.3.6 *Calibration Curve*

Transfer 0·500 g of pure iron into each of the six 250-ml PTFE beakers and dissolve according to the procedure specified in **4.3.1** and **4.3.2** using same amount of reagents. Add 0, 2·5, 5·0, 7·5, 10·0 and 12·5 ml of standard silicon solution (1ml = 20 µg Si) to each beaker and continue to follow rest of the procedure as specified in **4.3.3**. Prepare the calibration graph by plotting the net absorbance values against the silicon concentration in µg/ml, in the prepared solution.

NOTE — Blank solution and compensating solution are not necessary for the test solution as both are corrected via zero solution of standard silicon solution.

4.3.7 *Calculation*

Convert the corrected absorbance of each test solution and blank solution into µg/ml by the calibration graph.

Calculate silicon content as:

$$\text{Silicon, percent by mass} = \frac{(A - B)}{4\,M}$$

where

A = µg/ml of silicon in test solution (corrected for its compensating solution);

B = µg/ml of silicon in blank solution (corrected for its compensating solution); and

M = mass, in g, of sample.

4.3.8 *Reproducibility* — ± 0·005 at 0·03 percent level

± 0·01 at 0·05 percent level.

Bureau of Indian Standards

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Revision of Indian Standards

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