IS 228 (Part 8) : 1989
(Reaffirmed 1994)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 8 DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD (FOR SILICON 0·05 TO 5·00 PERCENT)

(Third Revision)

भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 8 गुरुत्वमापी पद्धति द्वारा सिलीकॉन ज्ञात करना (0·05 से 5·00 प्रतिशत सिलीकॉन के लिए)

(तीसरा पुनरीक्षण)

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BUREAU OF INDIAN STANDARDS
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Price Group 1

FOREWORD

This Indian Standard (Part 8) (Third Revision) was adopted by the Bureau of Indian Standards on 24 November 1989, 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, alongwith 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 which are being covered in separate standard. 14 parts have already been issued covering only chemical analysis of steels.

This standard IS 228 (Part 8) was published in 1975. In this revision the limit for determination of silicon has been modified as 0·05 to 5·00 percent in place of greater than or equal to 0·1 percent and the reproducibility of the method has also been incorporated.

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

Indian Standard

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PART 8 DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD (FOR SILICON 0.05 TO 5·00 PERCENT)

(Third Revision)

1 SCOPE

1.1 This standard (Part 8) covers the gravimetric method for determination of silicon (0·05 to 5·00 percent) in plain carbon and alloy steels including high speed steels containing tungsten.

2 SAMPLING

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

3 QUALITY OF REAGENTS

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

4 DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD

4.1 Outline of the Method

After dissolution of the sample, silicic acid is dehydrated by fuming with sulphuric acid. The solution is filtered, and silica is ignited, weighed, and then volatilized with hydroflouric acid. The residue is ignited and weighed; the loss in weight represent silica.

4.2 Reagents

4.2.1 *Mixed Acids*

Add 450 ml of concentrated nitric acid to 500 ml water and cool. To another 500 ml of water add 250 ml of concentrated sulphuric acid and cool. Mix both the dilute acids.

4.2.2 *Dilute Hydrochloric Acid*, 1 : 1, 3 : 1 and 1 : 20 (*v/v*).

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

4.2.4 *Hydroflouric Acid, O*, 40 percent.

4.2.5 *Concentrated Nitric Acid*, rd = 1·42 (conforming to IS 264 : 1976).

4.2.6 *Perchloric Acid*, 70 percent.

4.2.7 *Concentrated Sulphuric Acid*, rd = 1·84 (conforming to IS 266 : 1977).

4.2.8 *Dilute Nitric Acid*, 1 : 1 (*v/v*).

4.2.9 *Tartaric Acid*, 40 percent (*m/v*).

4.3 Procedure

4.3.1 *For Plain Carbon Steels*

Take 5·0 g of sample containing up to 0·1 percent silicon content, 2·5 g of sample for silicon content up to 1 percent and 1 g of sample for silicon content up to 5 percent, in a 300 ml porcelain casserole. Add 30 ml of mixed acid to dissolve the sample. Heat until nitrous fumes are expelled and continue heating till SO_3 fumes are evolved. Cool and add 50 ml of dilute hydrochloric acid (1 : 1) and heat again. Cool, dilute to 100 ml with warm water and add filter paper pulp. Stir well and filter immediately through medium textured filter paper. Wash the residue with hot dilute hydrochloric acid (1 : 20) and then with hot water till residue is free from chloride (check the solution for presence of chloride by 0·5 percent $AgNO_3$ solution). Transfer the residue and filter paper to a platinum crucible and dry at 110°C. Heat, char and ignite at 1 050°C for 30 minutes. Cool in a desiccator and weigh. Add 1-2 drops of dilute sulphuric acid to moisten the residue and then add 3-5 ml of hydrofluoric acid. Evaporate to dryness and ignite at 1 050°C to constant mass.

4.3.2 *For Steel Containing High Chromium*

Take 1 g of sample in a 500 ml beaker. Add 40 ml of dilute hydrochloric acid 3 : 1 and heat gently till reaction ceases. Add 4-5 ml of concentrated nitric acid and heat gently to expel nitrous fumes. Add 10 ml of perchloric acid and reflux for 15 minutes after perchloric acid fumes are evolved. Cool, add 50 ml of hot dilute hydrochloric acid (1 : 1). Follow rest of the procedure as given under **4.3.1** starting from addition of 50 ml of dilute hydrochloric acid (1 : 1).

4.3.3 *For High Speed Steels Containing Tungsten*

Take 2 g of sample in a beaker and dissolve in 50 ml of dilute sulphuric acid (1 : 1). Add 10 ml of dilute nitric acid and evaporate to copius fumes. Add 40 ml of dilute hydrochloric acid (1 : 1), and 70 ml of tartaric acid

(40 percent) and heat to dissolve the salts. Filter through medium textured filter paper and wash with dilute hydrochloric acid (1 : 20). Ignite the residue at 1 050°C in a platinum crucible, weigh and then moisten the residue with dilute sulphuric acid (1 : 1). Add 2 to 3 ml of hydrofluoric acid and evaporate to dryness. Reignite the residue at 750°C, cool and weigh.

4.3.4 *For High Silicon Steels*

Dissolve 1 g of the sample in 40 ml of dilute hydrochloric acid (1 : 1). Add 20 ml of concentrated nitric acid and 20 ml of perchloric acid and fume. Follow rest of the procedure as given in **4.3.1**.

4.3.5 *Blank*

Carry out a reagent blank using the procedure adopted for the determination.

4.4 Calculation

$$\text{Silicon, percent by mass} = \frac{(A - B) \times 46{\cdot}75}{C}$$

where

A = mass in g of silica obtained i sample,

B = mass in g of silica obtained i blank, and

C = mass in g of sample taken.

4.5 Reproducibility

± 0·02 at 0·5 percent level

± 0·05 at 2·0 percent level

± 0·07 at 4·0 percent level

Bureau of Indian Standards

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

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Amendments Issued Since Publication

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