BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 6 DETERMINATION OF SILICON BY GRAVIMETRIC METHOD (FOR SILICON 0.1 TO 6.0 PERCENT)

[*First Revision* of IS 12308 (Part 6)]

ICS 77.080.10

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34	Last date of comments **16 March 2023**

FOREWORD

This draft Indian Standard (Part 6) (First Revision) subject to its finalization, is to be adopted by the Bureau of Indian Standards on recommendation of the Methods of Chemical analysis of Metals Sectional Committee and approval of the Metallurgical Engineering Division Council.

Chemical analysis of cast iron and pig iron was covered in IS 228 'Methods of Chemical analysis of pig iron, cast iron and plain carbon and low alloy steels (*revised*)'. During its second revision it was decided that a comprehensive series should be prepared for chemical analysis of all type of steels and the other covering the chemical analysis of cast iron and pig iron. Accordingly IS 228 on revision was published in several parts covering chemical analysis of various steels only and a separate series of standards under IS 12308 is being published for chemical analysis of cast iron and pig iron.

This standard was first published in 1991 in different parts covering methods for chemical analysis of cast iron and pig iron. This standard (Part 6) covers determination of silicon by gravimetric method (for silicon 0.1 to 6.0 percent). The method given in this part for determination of silicon had been updated on the basis of experience gained.

The other parts in the series are:

Part 1	Determination of total carbon by thermal conductivity method
Part 2	Determination of sulphur by iodimetric titration method
Part 3	Determination of manganese by periodate spectrophotometric method
Part 4	Determination of total carbon, graphitic carbon and combined carbon by gravimetric method
Part 5	Determination of phosphorus by Alkalimetric method (for phosphorus 0.01 to 0.50 percent)
Part 7	Determination of nickel by dimethylglyoxime (Gravimetric) method (for nickel 0.5 to 36 percent)

Part 8	Determination of chromium by persulphate oxidation method (for chromium 0.1 to 28 percent)
Part 9	Determination of molybdenum by thiocyanate (Spectrophotometric) method (for molybdenum 0.1 to 1.0 percent)
Part 10	Determination of manganese (up to 7.0 percent) by arsenite (Volumetric) method
Part 11	Determination of total carbon by the direct combustion volumetric method (for carbon 1.50 to 4.50 percent)
Part 12	Determination of copper by atomic absorption spectrometric method (for copper 0.01 to 0.5 percent)
Part 13	Determination of magnesium by atomic absorption spectrometric method (for magnesium upto 0.1 percent)
Part 14	Determination of titanium by hydrogen peroxide (Spectrophotometric) method (for titanium up to 0.25 percent)

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

In reporting the result of a test or analysis made in accordance with this standard, 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 FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 6 DETERMINATION OF SILICON BY GRAVIMETRIC METHOD (FOR SILICON 0.1 TO 6.0 PERCENT)

(First Revision)

1 SCOPE

This standard (Part 6) describes the gravimetric method for determination of silicon in the range of 0.1 to 6.0 percent in cast iron and pig iron.

2 REFERENCE

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:

IS No.	*Title*
IS 1070 : 1992	Reagent grade water – Specification (*third revision*)

3 SAMPLING

The sample shall be drawn and prepared as prescribed in the relevant Indian Standard.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070) shall be employed in the test.

5 DETERMINATION OF SILICON BY GRAVIMETRIC METHOD

5.1 Outline of the Method

Sample is dissolved, silicic acid is dehydrated and silica is determined after hydrofluorization.

5.2 Reagents

5.2.1 *Dilute Nitric Acid* – 2:3 (*v/v*) and 1:2 (*v/v*).

5.2.2 *Dilute Hydrochloric Acid* – 1:1 (*v/v*).

5.2.3 *Perchloric Acid* – 70 percent (*v/v*).

5.2.4 *Tartaric Acid* – 20 percent (*m/v*).

5.2.5 *Dilute Sulphuric Acid* – 20 percent (*v/v*).

5.2.6 *Hydrofluoric Acid* – 40 percent (*v/v*).

5.3 Procedure

5.3.1 Transfer 0.500 to 2.000 g of sample (depending upon the silicon content) to a 400-ml tall-form beaker covered with a watch glass and dissolve in 20 ml of nitric acid (2:3 *see* **5.2.1**). When the violent reaction has ceased, add 20 ml of dilute hydrochloric acid (1:1 *see* **5.2.2**). Heat for a minute or so. Cool and add 20 ml perchloric acid. Evaporate the solution to fumes for 15 to 20 minutes at such a rate that the perchloric acid refluxes on the sides of the beaker.

5.3.2 Cool the solution and add 100 ml of hot water (40 to 50 °C), boil gently for two to three minutes till the iron salts dissolve.

NOTE – If the sample portion contains chromium (more than 100 mg) add 1 ml of tartaric acid solution for each 25 mg of chromium.

5.3.3 Add paper pulp to the solution and filter through medium textured filter paper, being careful to remove adhering particles from the beaker by rubber tipped glass rod. Wash the residue thoroughly with hot dilute hydrochloric acid (1:1) and finally with hot water (5-6 times) till free from chloride.

NOTE – Test the filtrate with 0.5 percent it silver nitrate solution.

5.3.4 Transfer the residue and the paper in a platinum crucible. Heat at 600 °C until the carbon is oxidized. Finally ignite the residue at 1 000 to 1 050 °C for 30 minutes, cool in a desiccator and weigh (M_1).

5.3.5 Add sufficient dilute sulphuric acid (*see* **5.2.5**) to moisten the residue and then add 5 to 10 ml of hydrofluoric acid. Evaporate to dryness and then heat gradually until sulphuric acid is removed. Ignite at 1 000 to 1 050 °C for 5 to 10 minutes, cool in a desiccator and weigh (M_2).

5.3.6 Carry out a blank determination, following the same procedure as specified in **5.3.1** to **5.3.5** and using the same amount of reagents.

5.4 Calculation

$$\text{Silicon, percent by mass} = \frac{(A - B) \times 46.72}{C}$$

where

$A = (M_1\text{-}M_2)$ = mass, in g, of silica obtained from the sample,

B = mass, in g, of silica obtained from the blank, and

C = mass, in g, of sample taken.

5.5 Reproducibility

± 0.002 at 0.2 percent Silicon,

± 0.01 at 4 percent Silicon, and

± 0.02 at 6 percent Silicon.