भारतीय मानक **IS 12308 (Part 6) : 2024**
Indian Standard

ढलवाँ लोहा और कच्चा लोहा के रासायनिक विश्लेषण की पद्धतियाँ

भाग 6 सिलिकॉन पद्धति द्वारा ग्रेवीमेट्रिक का निर्धारण (0.1 प्रतिशत से 6.0 प्रतिशत सिलिकॉन के लिए)

(पहला पुनरीक्षण)

Methods for Chemical Analysis of Cast Iron and Pig Iron

Part 6 Determination of Silicon by Gravimetric Method (for Silicon 0.1 Percent to 6.0 Percent)

(First Revision)

ICS 77.080.10

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FOREWORD

This Indian Standard (Part 6) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical analysis of Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1991. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

This standard is published in different parts covering methods for chemical analysis of cast iron and pig iron are as listed below. This standard (Part 6) covers determination of silicon by gravimetric method (for silicon 0.1 to 6.0 percent).

The other parts in the series are:

Part 1	Determination of total carbon by thermal conductivity method (for carbon 1.00 percent to 4.50 percent)
Part 2	Determination of sulphur by iodimetric titration method by iodimetric titration after combustion (for sulphur 0.005 percent to 0.25 percent)
Part 3	Determination of manganese by periodate spectrophotometric method (for manganese 0.1 percent to 2.5 percent)
Part 4	Determination of total carbon, graphitic carbon and combined carbon by gravimetric method
Part 5	Determination of phosphorus (0.01 percent to 0.50 percent) by alkalimetric method
Part 7	Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel 0.5 percent 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 percent 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)

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

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*)’.

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 6 DETERMINATION OF SILICON BY GRAVIMETRIC METHOD (FOR SILICON 0.1 PERCENT 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 percent to 6.0 percent in cast iron and pig iron.

2 REFERENCE

The standards given below contains provisions which through reference in this text, constitutes provisions of this standard. At the time of publication the edition indicated was valid. This standard is subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent edition of this standard:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth 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 g 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 min to 20 min 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 °C 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 times to 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 °C to 1 050 °C for 30 min, 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 ml to 10 ml of hydrofluoric acid. Evaporate to dryness and then heat gradually until sulphuric acid is removed. Ignite at 1 000 °C to 1 050 °C for 5 min to 10 min, 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

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

where

A = (M_1 – 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.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

Organization	*Representative(s)*
CSIR - National Metallurgical Laboratory, Jamshedpur	DR SANCHITA CHAKRAVARTY **(*Chairperson*)**
Arcelor Mittal Nippon Steel, Mumbai	SHRI MANOJ GUPTA SHRI KIRIT TAILOR (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	MISS SANJUKTA A. KUMAR SHRI M. V. RANA (*Alternate*)
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Defence Metallurgical Research Laboratory, Ministry of Defence, Hyderabad	SHRI S. S. KALYAN KAMAL
Directorate General of Quality Assurance, Ministry of Defence, New Delhi	SHRI KESAVAMOORTHY M. SHRI E. SUMAN. KUMAR (*Alternate*)
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Organization	*Representative(s)*
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TRL Krosaki Refractories Limited, Belpahar	SHRI S. K. SUBUDHI
BIS Directorate General	SHRI SANJIV MAINI, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (METALLURGICAL ENGINEERING) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI ASHISH PRABHAKAR WAKLE
SCIENTIST 'D'/JOINT DIRECTOR
(METALLURGICAL ENGINEERING), BIS

Bureau of Indian Standards

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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