भारतीय मानक **IS 12308 (Part 2) : 2024**

Indian Standard

ढलवाँ लोहा और कच्चा लोहा के रासायनिक विश्लेषण की पद्धतियाँ

भाग 2 दहन के बाद आयोडिमेट्रिक अनुमापन द्वारा सल्फर का निर्धारण (0.005 प्रतिशत से 0.25 प्रतिशत सल्फर के लिए)

(पहला पुनरीक्षण)

Methods for Chemical Analysis of Cast Iron and Pig Iron

Part 2 Determination of Sulphur by Iodimetric Titration after Combustion (For Sulphur 0.005 Percent to 0.25 Percent)

(First Revision)

ICS 77.080.10

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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 2) (First Revision) was adopted by the Bureau of Indian Standards after the finalized by the Methods of Chemical analysis of Metals Sectional Committee had been approved by of the Metallurgical Engineering Division Council.

This standard was first published in 1987. 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 2) covers determination of sulphur by iodimetric titration after combustion (for sulphur 0.005 percent to 0.25 percent).

The other parts in the series are:

Part 1	Determination of total carbon by thermal conductivity 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 percent to 0.50 percent)
Part 6	Determination of silicon (for silicon 0.1 to 6.0 percent)
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 percent to 28 percent)
Part 9	Determination of molybdenum by thiocyanate (spectrophotometric) method (for molybdenum 0.1 percent 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 percent 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, 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*)’.

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 2 DETERMINATION OF SULPHUR BY IODIMETRIC TITRATION AFTER COMBUSTION (FOR SULPHUR 0.005 PERCENT TO 0.25 PERCENT)

(First Revision)

1 SCOPE

This standard (Part 2) covers method for determination of sulphur in cast iron and pig iron in the range of 0.005 percent to 0.25 percent.

2 REFERENCES

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. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standard:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 6226 (Part 2) : 1987	Recommendations for apparatus for chemical analysis of metals: Part 2 Determination of sulphur by direct combustion

3 QUALITY OF REAGENTS

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

4 SAMPLING

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

4.2 The sample is to be cleaned with analytical grade organic solvent (like acetone, benzene or ether) by washing it thrice and dried in an air oven at 100 °C ± 5 °C.

5 DETERMINATION OF SULPHUR IN CAST IRON AND PIG IRON BY THE IODIMETRIC TITRATION AFTER COMBUSTION

5.1 Outline of the Method

The sample is burnt in a stream of oxygen. The sulphur dioxide formed is absorbed in an acidified starch-iodide solution and continuously titrated with standard potassium iodate solution.

5.2 Reagents

5.2.1 *Oxygen* (O_2) — 99.5 percent minimum purity

5.2.2 *Ascarite of Soda Lime* — 0.80 mm to 2.00 mm

5.2.3 *Magnesium Perchlorate* [$Mg(ClO_4)_2$] — 0.80 mm to 2.00 mm

5.2.4 *Ceramic Boats/Crucibles*
Ceramic boats or crucibles of the precise dimensions which may be accommodated in the combustion tube/induction furnace.

5.2.4.1 Pre-ignite the boats/crucibles in stream of air or oxygen in a furnace at 1 100 °C for 15 min and store in a desiccator.

5.2.5 *Fluxes* — Low sulphur copper, tin or iron

5.2.6 *Dilute Hydrochloric Acid* — 1 : 30 (*v/v*)

5.2.7 *Starch Solution*

Transfer 9 g of soluble starch to a 50 ml beaker, add 100 ml water, stir until a smooth paste is obtained. Pour the mixture slowly into 500 ml of boiling water. Cool, add 15 g of potassium iodide and stir until it dissolves. Dilute to 1 litre and mix.

5.2.8 *Standard Potassium Iodate Solution*

Dissolve 0.222 5 g of potassium iodate in 900 ml of water containing 1.0 g of sodium hydroxide and dilute to 1 000 ml in a volumetric flask.

5.2.8.1 *Standard potassium iodate solution*

Transfer 200 ml of potassium iodate solution (5.2.8) to a 1 000 ml volumetric flask. Dilute to the mark with water and mix. Standardize the solutions (5.2.8) and 5.2.8.1 daily against standard cast iron samples of similar composition after combustion and following the procedure as given in 5.4 and find out the sulphur equivalent for 1 ml of the potassium iodate solutions.

5.3 Apparatus

The apparatus recommended in IS 6226 (Part 2) may be used.

5.4 Procedure

5.4.1 Switch on the furnace, allow it to attain a temperature of 1 425 °C to 1 450 °C. All the while passing oxygen through the apparatus at a rate of approximately 1 000 ml/min to 1 500 ml/min. Take 50 ml to 70 ml of dilute hydrochloric acid and 2 ml of starch solution in the absorption vessel. Add potassium iodate solution (5.2.8.1) from a burette to obtain a blue colour, the intensity of which is to be taken as the end point of the final titration. Refill the burette and adjust to zero mark.

5.4.2 Weigh 1 000 g of the sample, for sulphur content below 0.06 percent and 0.500 g for above 0.06 percent, transfer to the pre-ignited boat or crucible, cover the sample with 1 g of the flux. Introduce the boat in the centre of the combustion zone of the combustion tube and close the tube. In the case of induction heating, place the crucible on the pedestal post of the induction furnace, raise it to the combustion position and switch on the furnace.

5.4.3 As the oxygen gas stream flows through the absorption solution, the blue colour fades. Titrate continuously with potassium iodate solution (5.2.8) in the case of sulphur content greater than 0.02 percent and (5.2.8.1) for sulphur content less than 0.02 percent at such a rate as to maintain the initial intensity of the blue colour. At the end of the combustion period (which is around 5 min), there would be no further decrease in the intensity of the blue colour. Take the burette reading and note the volume of titrant added.

NOTE — Do not allow the solution to become colourless at the time during the titration to avoid possible loss of sulphur dioxide.

5.4.4 *Blank*

Make a blank determination, following the same procedure and using the same amount of all reagents except the addition of sample.

NOTE — In some cases, the boats or crucibles may not give out their inherent sulphur, unless a sample is combusted. This may be verified by combusting a sulphur free sample in the boat.

5.4.5 *Calculation*

Calculate the percentage of sulphur as follows:

$$\text{Sulphur, percent} = \frac{(A - B)\,C}{D} \times 100$$

where

A = volume, in ml, of standard potassium iodate solution required for titration of the sample;

B = volume, in ml, of standard potassium iodate solution required for titration of the blank;

C = sulphur, in g/ml, equivalent of the potassium iodate solutions (5.2.8 and 5.2.8.1) calculated by titration with standard sample of cast iron; and

D = mass, in g, of the sample taken.

5.4.6 *Reproducibility* — ± 0.002 percent

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*)
CSIR - National Metallurgical Laboratory, Jamshedpur	DR ASHOK K. MOHANTY
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*)
Geological Survey of India, New Delhi	SHRI NITIN PURUSHOTTAM SHRIMATI SANJUKTA DEY PAL (*Alternate*)
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Indian Metals and Ferro Alloys Limited, Bhubaneswar	SHRI DINESH KUMAR MOHANTY
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TRL Krosaki Refractories Limited, Belpahar	SHRI S. K. SUBUDHI

Organization	*Representative(s)*
Tata Steel Limited, Kolkata	DR JATIN MOHAPATRA DR RAVIKRISHNA CHATTI (*Alternate*)
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Member Secretary
SHRI ASHISH PRABHAKAR WAKLE
SCIENTIST 'D'/JOINT DIRECTOR
(METALLURGICAL ENGINEERING), BIS

Bureau of Indian Standards

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Review of Indian Standards

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