BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

Draft 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 TO 0.25 PERCENT)

[*First Revision* of IS 12308 (Part 2)]

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 2) (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 : 1959 'Methods of chemical analysis of pig iron, cast iron and plain carbon and low alloy steels (*revised*)'. During the second revision of this standard, it was decided that a comprehensive series on above standard should be prepared only for chemical analysis of steels, and chemical analysis of cast iron and pig iron be covered in a separate standard. Accordingly, IS 228, in its various parts, was published for chemical analysis of steels.

This standard was first published in 1987 in different parts covering methods for chemical analysis of cast iron and pig iron. This standard (Part 2) covers determination of sulphur by iodimetric titration after combustion (for sulphur 0.005 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 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 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 2 DETERMINATION OF SULPHUR BY IODIMETRIC TITRATION AFTER COMBUSTION (FOR SULPHUR 0.005 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 to 0.25 percent.

2 REFERENCE

The Indian Standards listed 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 the standard indicated below:

IS No	*Title*
IS 1070 : 1992	Reagent grade water – Specification (*third 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 analar grade organic solvent (like acetone, benzene or ether) by washing it thrice and dried in an air oven at 100 ± 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* — 0.80-2.00 mm.

5.2.3 *Magnesium perchlorate* [$Mg(ClO_4)_2$] – 0.80-2.00 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 minutes 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 to 1 450 °C. All the while passing oxygen through the apparatus at a rate of approximately 1 000 to 1 500 ml/minute. Take 50 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 equivalent of the potassium iodate solutions (**5.2.8** and **5.2.8.1**) in g/ml, 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.