भारतीय मानक प्रारूप

ढलवां लोहे और कच्चे लोहे के रासायनिक विश्लेषण की पद्धतियाँ

भाग 3 XXXX

(*पहला पुनरीक्षण*)

Draft Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 3 DETERMINATION OF MANGANESE BY PERIODATE SPECTROPHOTOMETRIC METHOD (FOR MANGANESE 0.1 TO 2.5 PERCENT)

(First Revision)

ICS 77.080.10

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

XXXX **2022** **Price Group**

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This draft Indian Standard (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.

Method for determination of manganese in cast iron and pig iron by bismuthate method and persulphate oxidation method were given in IS 228 : 1959 'Methods of chemical analysis of pig iron, cast iron and plain carbon and low alloy steels (*revised*)'. IS 228 : 1959 was again revised to make it comprehensive in respect of steel analysis. To cover chemical analysis of cast iron and pig iron, it was decided that a separate standard be published in series. This standard is one of the series of Indian Standards on methods of chemical analysis of cast iron and pig iron, determination of manganese by periodate spectrophotometric method. With the publication of this standard, the bismuthate method will be superseded.

This standard was first published in 1987 in different parts covering methods for chemical analysis of cast iron and pig iron. This standard (Part 3) covers determination of manganese by periodate spectrophotometric method (for manganese 0.1 to 2.5 percent).

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 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*)’. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Draft Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 3 DETERMINATION OF MANGANESE BY PERIODATE SPECTROPHOTOMETRIC METHOD
(FOR MANGANESE 0.1 TO 2.5 PERCENT)

(First Revision)

1 SCOPE

This standard (Part 3) covers the method for determination of manganese in cast iron and pig iron in the range of 01 to 2.5 percent.

2 REFERENCE

2.1 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*
264 : 2005	Nitric acid – Specification (*third revision*)
1070 : 1992	Reagent grade water – Specification (*third revision*)

3 SAMPLING

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

4 QUALITY OF REAGENT

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070)

5 DETERMINATION OF MANGANESE IN CAST IRON AND PIG IRON BY PERIODATE SPECTROPHOTOMETRIC METHOD.

5.1 Outline of the Method

After dissolution of the sample in sulphuric/phosphoric acid mixture and nitric acid, the solution is fumed with perchloric acid. Manganese is oxidized to per manganic acid by potassium periodate. Absorbance of the solution is measured at 545 nm.

5.2 Reagents

5.2.1 *Sulphuric-phosphoric acid mixture* – Add 100 ml of concentrated sulphuric acid (rd 1.84) arid 150 ml of phosphoric acid (rd 1.75) to 600 ml of water. Cool and dilute to 1 litre with water.

5.2.2 *Concentrated nitric acid* — (rd 1.42) (Conforming to IS 264)

5.2.3 *Perchloric acid* — 70 percent.

5.2.4 *Dilute perchloric acid* – 1:99 (*v/v*).

5.2.5 *Potassium periodate solution*

Dissolve 7.5 g of potassium periodate in 200 ml of dilute nitric acid (1:1) and add 400 ml of phosphoric acid; cool, and dilute to 1 litre.

5.2.6 *Standard manganese solution* (1 ml = 0.01 mg Mn)

Prepare as described in Method A and B:

Method A (*Preparation from manganese metal*) — Take 0.1 g of manganese metal (purity 99.8 percent, *Min*) in a beaker and add 10 ml of dilute nitric acid (1:1). Heat gently until dissolution IS complete and brown fumes are expelled. Cool, transfer to 1 litre volumetric flask, dilute to volume and mix. Take 10 ml of the solution and dilute to 100 ml.

Method B (*Preparation from potassium permanganate*) — Dissolve 3.2 g of potassium permanganate ($KMnO_4$) in 1 litre of water. Let stand in the dark for 2 weeks. Filter without washing through a Gooch crucible. Avoid contact with rubber or other organic material. Store in a dark coloured glass stoppered bottle.

5.2.6.1 *Standardization for method B* — Dry a portion of sodium oxalate at 105°C. Transfer 0.300 0 g of sodium oxalate to 500 ml beaker. Add 250 ml of dilute sulphuric acid (1:19), previously boiled for 10 to 15 minutes and then cooled to 27 ± 3°C and stir until the oxalate has dissolved. Add about 40 ml of potassium permanganate solution at a rate of 25 to 35 ml/min, while stirring slowly. Let stand until the pink colour disappears. Heat to 55 to 60°C and complete the titration by adding potassium permanganate solution until a faint pink colour persists for 30 seconds. Find the normality of the solution and adjust to 0.100 0 N.

Transfer 90.9 ml of 0.100 0 N potassium permanganate solution to 500-m1 beaker and add 10 ml of dilute sulphuric acid (1:1). Reduce the potassium permanganate solution by sulphurous acid and boil the solution until free of sulphur dioxide. Cool, and transfer to 1 litre volumetric flask and make up. Lake 10 ml of the solution and dilute to 100 ml.

5.3 Procedure

5.3.1 Take 1.00 g of sample in a 250-ml conical flask, add 50 ml of sulphuric-phosphoric acid mixture. Heat gently until action cesses. When sample is dissolved, oxidize with a few drone of concentrated nitric acid and add 10 ml of perchloric acid (*see* Note). Evaporate until white perchloric acid fumes are given off and keep at this temperature for 10 minutes.

NOTE – If the sample does not dissolve readily in acid mixture, add 5 ml each of concentrated hydrochloric acid and concentrated nitric acid to facilitate the dissolution.

5.3.2 After cooling, dilute to 60 to 70 ml with water and boil. If necessary; filter and wash with hot dilute perchloric acid. Collect the filtrate in 100-ml volumetric flask. Dilute to mark and mix well.

5.3.3 Take a suitable aliquot from the above solution (containing 0.1 to 1 mg of manganese) in a 250-ml conical flask and bring it to boil. Add 5 ml of acid mixture and 10 ml of potassium periodate solution and boil at 90°C for 10 minutes. Cool to ambient temperature. Transfer to 10-ml volumetric flask and dilute to mark with water (*see* Note) and mix. Measure the absorbance at 545 nm against a reagent blank.

NOTE – All water used for dilution should be pretreated with potassium periodate.

5.3.4 *Blank*

Carry out a blank using the same quantity of the reagents used.

5.3.5 *Calibration curve*

Transfer 0, 1.0, 2.0, 4.0, 6.0, 8.0 to and 10.0 ml of standard manganese solution (1 ml = 0.01 mg Mn) to seven 100-m1 volumetric flasks and proceed according to **5.3.3**. Draw a calibration curve of absorbance values against milligrams of manganese in the various aliquots.

5.3.6 *Calculation*

Convert the spectrophotometric reading of the sample taken under **5.3.3** to milligrams of manganese by means of calibration curve and calculate the percentage of manganese as follows:

$$\text{Manganese, percent} = \frac{A}{B} \times 0.1$$

where

A = mass in mg of manganese found in the aliquot of the solution, and

B = mass in g of sample represented by aliquot of the solution taken.

5.3.7 *Reproducibility*

±0.01 percent for manganese content up to 0.3 percent,

± 0.02 percent for manganese content between 0.7 to 1 percent,

± 0.06 percent for manganese content between 0.7 to 1 percent,

± 0.08 percent for manganese content between 1 to 1.6 percent, and

± 0.04 percent for manganese content between 1.6 to 2.5 percent.