भारतीय मानक प्रारूप

ढलवां लोहे और कच्चे लोहे के रासायनिक विश्लेषण की पद्धतियाँ

भाग 10 आर्सेनाइट, आयतनी पद्धति से मैंगनीज (7.0 प्रतिशत तक) ज्ञात करना

(*पहला पुनरीक्षण*)

Draft Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 10 DETERMINATION OF MANGANESE (UP TO 7.0 PERCENT) BY ARSENITE (VOLUMETRIC) METHOD

(*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 (Part 10) (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 its second revision it was decided that a comprehensive series should be prepared for chemical analysis of all types 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 10) covers determination of manganese (up to 7.0 percent) by arsenite (volumetric) method. In this part the bismuthate method for determination of manganese (in the absence of cobalt) in cast iron and pig iron had been replaced by arsenite volumetric method for determination of manganese (in absence of cobalt) in plain cast iron and pig iron.

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 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 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 10 DETERMINATION OF MANGANESE (UP TO 7.0 PERCENT) BY ARSENITE (VOLUMETRIC) METHOD

(*First Revision*)

1 SCOPE

1.1 This standard (Part 10) prescribes the method for determination of manganese (up to 7.0 percent) in plain cast iron and pig iron containing less than 0.5 percent of chromium and no cobalt content.

1.2 This method is not applicable to alloy cast iron.

2 SAMPLING

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

3 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

4 METHOD

4.1 Outline of the Method

The sample is dissolved in acid mixture (phosphoric acid, sulphuric acid and nitric acid). Manganese is oxidised by ammonium persulphate in the presence of silver nitrate and titrated with standard sodium arsenite solution.

4.2 Reagents

4.2.1 *Acid Mixture*

To 400 ml of water, cautiously, add 100 ad of sulphuric acid (rd = 1.84), 125 ml of phosphoric acid (rd= 1.75) and 250 ml of nitric acid (rd = 1.42). Cool, dilute to 1 litre and mix.

4.2.2 *Silver Nitrate Solution* 0.2 percent (*m/v*).

4.2.3 *Sodium Chloride Solution* 0.2 percent (*m/v*).

4.2.4 *Ammonium Persulphate Solution 10 percent* (*m/v*), freshly prepared.

4.2.5 *Standard Iron*, having same manganese content as the sample.

4.2.6 *Sodium Arsenite* ($NaAsO_2$), solid.

4.2.6.1 *Standard arsenite solution*

Dissolve 1.05 g of sodium arsenite in 2 litres of water containing 2 g of sodium bicarbonate. Standardise this solution (*see* **4.3**) with an iron sample of known manganese (**4.2.5**) content.

4.3 Procedure

4.3.1 Transfer 0.100 g sample to a 250-m1 conical flask. Add 30-ml of acid mixture. Heat gently to dissolve the sample and boil off nitrous fumes. Dilute to 50-m1 with water, filter through a medium tenured filter paper and wash 3-4 times with hot water. To the filtrate, add 10 ml of silver nitrate solution, followed by 20 ml of ammonium persulphate solution. Boil for 20 s, remove from hot plate and cool rapidly to room temperature. Add 10 ml of sodium chloride solution and titrate immediately with standard sodium arsenite solution shaking vigorously until the pink colour is just discharged.

Take 0.100 g of a standard iron sample of a known manganese content and proceed as above. Both standard and sample are to be titrated under identical operative conditions.

NOTE – It is necessary to use standard iron sample having same manganese content as the sample under analysis.

4.3.2 Run a blank using all the reagents, through the procedure specified in **4.3.1**.

4.4 *Calculation*

$$\text{Manganese, percent by mass} = \frac{V_1 - V_2}{V_3 - V_2} \times \text{percentage of manganese in the standard}$$

where

V_1 = volume, in ml, of sodium arsenite used for sample;

V_2 = volume, in ml, of sodium arsenite used for blank; and

V_3 = volume me, in ml, of sodium arsenite used for standard.