

भारतीय मानक प्रारूप
इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 2 XXXX

(चौथा पुनरीक्षण)

Draft Indian Standard

METHODS FOR
CHEMICAL ANALYSIS OF STEELS

PART 2 DETERMINATION OF MANGANESE IN PLAIN-CARBON AND LOW ALLOY
STEELS BY ARSENITE METHOD

(Fourth Revision)

ICS 77.080.20

© BIS 2022
BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This draft Indian Standard (Part 2) (Fourth 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.

This standard was first published in 1952 and subsequently revised in 1959, 1972 and 1987, covered the chemical analysis of plain carbon and low alloy steels, along with pig iron and cast iron. It was revised again to make it comprehensive in respect of steel analysis and to exclude pig iron and cast iron which were being covered in separate standards. During its second revision the standard has been split up in several parts.

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

Photometric method for determination of manganese up to 5 percent has been covered in IS 228 (Part 12).

This part covers method for the determination of manganese from 0.1 to 1.5 percent in plain-carbon and low alloy steels by arsenite method. The other parts of this series are:

- Part 1 Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent)
- Part 3 Determination of phosphorus by alkalimetric method
- Part 4 Determination of total carbon by gravimetric method (for carbon greater than or equal to 0.1 percent)
- Part 5 Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel greater than or equal to 0.1 percent)
- Part 6 Determination of chromium by persulphate oxidation method (for chromium ≥ 0.1 percent)
- Part 7 Determination of molybdenum by alpha benzoinoxime method (for molybdenum 1 percent and not containing tungsten)
- Part 8 Determination of silicon by gravimetric method (for silicon 0.05 to 5.00 percent)
- Part 9 Determination of sulphur in plain carbon steels by evolution method (for sulphur 0.01 to 0.25 percent)

- Part 10 Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum 0.01 to 1.5 percent)
- Part 11 Determination of silicon by reduced molybdsilicate spectrophotometric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent)
- Part 12 Determination of manganese by periodate spectrophotometric method in plain carbon, low alloy and high alloy steels (for manganese 0.01 to 5.0 percent)
- Part 13 Determination of arsenic
- Part 14 Determination of carbon by thermal conductivity method (for carbon 0.005 to 2.000 percent)
- Part 15 Determination of copper by thiosulphate iodide method (for copper 0.05 to 5 percent)
- Part 16 Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent)
- Part 17 Determination of nitrogen by thermal conductivity method
- Part 18 Determination of oxygen by instrumental method
- Part 19 Determination of nitrogen by steam distillation
- Part 20 Determination of carbon and sulphur by infrared absorption method
- Part 21 Determination of copper by spectrometric method (for copper 0.02 to 0.5 percent)
- Part 22 Determination of total hydrogen in steel by thermal conductivity method (hydrogen 0.1 ppm to 50 ppm)
- Part 23 Determination of total nitrogen in steel by optical emission spectrometer (nitrogen 0.002 to 1.0 percent)
- Part 24 Determination of nitrogen in steel by inert gas fusion – Thermal conductivity method (nitrogen 0.001 to 0.2 percent)

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 STEELS

PART 2 DETERMINATION OF MANGANESE IN PLAIN-CARBON AND LOW ALLOY STEELS BY ARSENITE METHOD

(Fourth Revision)

1 SCOPE

1.1 This standard (Part 2) covers method for the determination of manganese in plain carbon and low alloy steels by arsenite method.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No</i>	<i>Title</i>
264 : 2005	Nitric acid – Specification (<i>third revision</i>)
1070 : 1992	Reagent grade water – Specification (<i>third revision</i>)

3 SAMPLING

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

4 QUALITY OF REAGENTS

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

5 DETERMINATION OF MANGANESE (0.1 TO 1.5 PERCENT) IN PLAIN CARBON AND LOW ALLOY STEELS BY THE ARSENITE METHOD (IN ABSENCE OF TUNGSTEN)

5.1 Outline of the Method

Manganese is oxidized, in presence of silver nitrate, to permanganic acid by ammonium persulphate and titrated with sodium arsenite solution.

5.2 Reagents

5.2.1 *Dilute Nitric Acid* – 1:2 (v/v).

5.2.2 *Phosphoric Acid* – 85 percent.

5.2.3 *Dilute Sulphuric Acid* – 1:4 (v/v).

5.2.4 *Concentrated Nitric Acid* – Relative density 1.42 (conforming to IS 264).

5.2.5 *Ammonium Persulphate Solution* – Freshly prepared, 10 percent (m/v).

5.2.6 *Silver Nitrate Solution* – 1 percent (m/v).

5.2.7 *Sodium Chloride Solution* – 1 percent (m/v).

5.2.8 *Standard Sodium Arsenite Solution*

Take 1.6 g of arsenious oxide in a 800-ml beaker. Add 10 g of sodium carbonate and 500 ml of water in the beaker and heat at low temperature until the solution is complete. Filter the solution through a filter pad in a bottle and make up the volume of the solution to 2 litres by addition of water. Shake the bottle vigorously. Standardize the arsenite solution as in **5.3** against 0.2 g of steel sample (having approximately similar composition as the sample under test) of known manganese content.

5.2.8.1 Adjust the strength of the sodium arsenite solution in such a way that each millilitre of the solution will be equivalent to 0.1 percent manganese when 0.2 g of sample is taken.

5.3 Procedure

5.3.1 *Dissolution*

5.3.1.1 *Plain carbon steel*

Take 0.2 g of an accurately weighed sample in a 250-ml conical flask. Add 10 ml of dilute nitric acid and 3 to 4 ml of phosphoric acid, and heat to dissolve the sample, boil to expel oxides of nitrogen and dilute to 100 ml.

5.3.1.2 *Low alloy steel*

Take 0.2 g of an accurately weighed sample in a 250-ml conical flask. Add 20 ml sulphuric acid and 2-3 ml of phosphoric acid. Heat until the reaction ceases. Add concentrated nitric acid drop by drop until the dissolution is complete, boil off nitrous fumes and dilute to 100 ml with water.

5.3.2 Add 20 ml of ammonium persulphate solution and boil. After few minutes of boiling, when the solution becomes clear, add 10 ml of silver nitrate and allow the colour of permanganic acid to develop. Boil to destroy the excess of persulphate. Cool quickly to room temperature.

5.3.3 Add 10 ml of sodium chloride solution to precipitate silver chloride. Titrate rapidly the permanganic acid with standard sodium arsenite solution until the pink colour is discharged.

6 CALCULATION

6.1 Calculate the manganese content of the steel as follows:

$$\text{Manganese, percent} = \frac{A \times B}{C} \times 100$$

where

A = volume of standard sodium arsenite solution used for titration,

B = manganese equivalent of standard sodium arsenite solution in g/ml, and

C = mass in g of sample.

6.2 Reproducibility

- a) ± 0.01 percent for manganese content below 0.06 percent,
- b) ± 0.02 percent for manganese content between 0.06 to 1 percent, and
- c) ± 0.03 percent for manganese above 1 percent.