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METHODS FOR CHEMICAL ANALYSIS OF STEELS

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

(*Third Revision*)

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

(Third Revision)

0. FOREWORD

0.1 This Indian Standard (Part 2) (Third Revision) was adopted by the Indian Standards Institution on 16 January 1987, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 228, which was issued as a tentative standard in 1952, and revised in 1959, covered the chemical analysis of pig iron, cast iron and plain carbon and low alloy steels. For the convenience it was decided to publish a comprehensive series on chemical analysis of steels including high alloy steels. Accordingly, revision of IS : 228 was taken-up again and new series on methods of chemical analysis of steels including high alloy steels was published in various parts as IS : 228 (Parts 1 to 13) (*see* Appendix A) covering separate method of analysis for each constituent in steels. However, IS : 228-1959* version has been retained for the analysis of pig iron and cast iron till a separate standard for analysis of pig iron and cast iron is published.

0.2.1 This revision of IS : 228 (Part 2)-1972† has been undertaken on the basis of experience gained during the implementation of the standard by the manufacturers and testing laboratories.

0.3 In this revision major modifications are:

a) modification of method for dissolution of low alloy steels, and

*Methods of chemical analysis of pig iron, cast iron and plain carbon and low-alloy steels (*revised*).

†Methods for chemical analysis of steels: Part 2 Determination of manganese in plain-carbon and low alloy steels by arsenite method (*second revision*).

b) inclusion of reproducibility of the method at the various levels of manganese content.

0.4 Photometric method for determination of manganese up to 2 percent has been covered in IS : 228 (Part 12)-1976*.

0.5 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 : 2-1960†.

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. SAMPLING

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

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water (*see* IS : 1070-1977)‡ shall be employed in the test.

4. DETERMINATION OF MANGANESE (0·1 TO 1·5 PERCENT) IN PLAIN CARBON AND LOW ALLOY STEELS BY THE ARSENITE METHOD (IN ABSENCE OF TUNGSTEN)

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

4.2 Reagents

4.2.1 *Dilute Nitric Acid* — 1 : 2 (*v*/*v*).

4.2.2 *Phosphoric Acid* — 85 percent.

4.2.3 *Dilute Sulphuric Acid* — 1 : 4 (*v*/*v*).

*Methods for chemical analysis of steels: Part 12 Determination of manganese by periodate (photometric) method in low and high alloy steels (for manganese up to 2 percent) (*second revision*).

†Rules for rounding off numerical values (*revised*).

‡Specification for water for general laboratory use (*second revision*).

4.2.4 *Concentrated Nitric Acid* — Relative density 1·42 (conforming to IS : 264-1976*).

4.2.5 *Ammonium Persulphate Solution* — Freshly prepared, 10 percent (*m*/*v*).

4.2.6 *Silver Nitrate Solution* — 1 percent (*m*/*v*).

4.2.7 *Sodium Chloride Solution* — 1 percent (*m*/*v*).

4.2.8 *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 **4.3** against 0·2 g of steel sample (having approximately similar composition as the sample under test) of known manganese content.

4.2.8.1 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.

4.3 Procedure

4.3.1 *Dissolution*

4.3.1.1 *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.

4.3.1.2 *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.

4.3.2 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.

*Specification for nitric acid (*second revision*).

4.3.3 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.

5. CALCULATION

5.1 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 = maganese equivalent of standard sodium arsenite solution in g/ml, and

C = mass in g of sample.

5.2 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.

APPENDIX A

(*Clause* 0.2)

IS : 228 Methods for chemical analysis of steels:

(Part 1)-1972 Determination of carbon by volumetric method (for carbon ⩾ 0·1 percent) (*second revision*)

(Part 2)-1972 Determination of manganese in plain carbon and low alloy steels by arsenite method (*second revision*)

(Part 3)-1972 Determination of phosphorus by alkalimetric method (*second revision*)

(Part 4)-1974 Determination of carbon by gravimetric method (for carbon ⩾ 0·1 percent) (*second revision*)

(Part 5)-1974 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel $\geq$ 0·5 percent) (*second revision*)

(Part 6)-1974 Determination of chromium by persulphate oxidation method (for chromium $\geq$ 0·5 percent) (*second revision*)

(Part 7)-1974 Determination of molybdenum by α-benzoinoxime method (for molybdenum > 1 percent) (*second revision*)

(Part 8)-1975 Determination of silicon by the gravimetric method (for silicon $\geq$ 0·1 percent) (*second revision*)

(Part 9)-1975 Determination of sulphur in plain carbon steels by evolution method (*second revision*)

(Part 10)-1976 Determination of molybdenum by thiocyanate (photometric) method (for molybdenum up to 1 percent) in low and high alloy steels (*second revision*)

(Part 11)-1976 Determination of silicon by photometric method in carbon steels and low alloy steels (for silicon 0·01 to 0·05 percent) (*second revision*)

(Part 12)-1976 Determination of manganese by periodate (photometric) method in low and high alloy steels (for manganese up to 2 percent) (*second revision*)

(Part 13)-1982 Determination of arsenic

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