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(Reaffirmed 1997)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 6 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION METHOD (FOR CHROMIUM ≥ 0·1 PERCENT)

(*Third Revision*)

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Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 6 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION METHOD (FOR CHROMIUM ≥ 0·1 PERCENT)

(Third Revision)

0. FOREWORD

0.1 This Indian Standard (Part 6) (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 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 6)-1974† 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) scope of the method has been modified by lowering the limit for determination of chromium from 0·5 to 0·1 percent;

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

†Methods for chemical analysis of steels: Part 6 Determination of chromium by persulphate oxidation method (for chromium ≥ 0·5 percent) (*second revision*).

b) only one method has been prescribed for the correction in the titration of chromium for dilution effect and colour interference; and

c) inclusion of reproducibility of the method at the various levels of chromium content.

0.4 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 6) covers the persulphate oxidation method for determination of chromium content of low alloy and high alloy steels containing more than or equal to 0·1 percent chromium. This method is not applicable for steels containing tungsten.

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. OUTLINE OF THE METHOD

4.1 After dissolution of the sample in dilute sulphuric acid and phosphoric acid mixture and further treated with nitric acid, chromium, manganese and (vanadium if present) are oxidized by ammonium persulphate in presence of silver nitrate as catalyst. Permanganic acid is then destroyed by dilute hydrochloric acid. Chromium is reduced by ferrous ammonium sulphate and excess of ferrous ammonium sulphate is back titrated with standard potassium permanganate solution.

5. REAGENTS

5.1 Phosphoric Acid — Sulphuric Acid Mixture — To 600 ml of water, add continuously 165 ml of concentrated sulphuric acid (rd = 1·84) and 132 ml of phosphoric acid (rd = 1·75). Mix, cool and dilute to 1 litre.

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

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

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

5.3 Silver Nitrate Solution — 0·5 percent (*m/v*). Dissolve 5 g of silver nitrate crystals in water and dilute to 1 litre.

5.4 Ammonium Persulphate Solution — Dissolve 15 g of ammonium persulphate in 100 ml of water. Use a freshly prepared solution.

5.5 Potassium Permanganate Solution — 1 percent (*m/v*).

5.6 Dilute Hydrochloric Acid — 1 : 3 (*v/v*). Dilute 250 ml of concentrated hydrochloric acid (rd = 1·16) to 1 litre.

5.7 Standard Ferrous Ammonium Sulphate Solution — Approximately 0·1 N. Dissolve 40 g of ferrous ammonium sulphate in sulphuric acid (5 percent) and dilute to 1 litre. Filter, if necessary, and keep in a stoppered glass bottle. Standardize against standard potassium permanganate solution (given under **5.8**) every time it is used.

5.8 Standard Potassium Permanganate Solution — Approximately 0·1 N. Dissolve 3·2 g of potassium permanganate crystals in 1 000 ml of water, stir and allow to stand in a closed vessel for 24 hours. Filter, through a sintered glass crucible and keep in an amber-coloured glass bottle. Standardize the solution as follows:

> Dissolve 0·134 g of sodium oxalate crystals, dried for 1 hour at 105°C, in 200 ml of dilute sulphuric acid (1 : 50). Heat to 70°C and titrate with potassium permanganate solution until one drop produces a permanent pink colouration. [1 ml of potassium permanganate solution (0·1 N) = 0·006 7 g of sodium oxalate].

6. PROCEDURE

6.1 Take 2 g of sample (for chromium less than 2 percent) and 0·2 to 0·5 g of sample for high alloy steels in a wide mouth conical flask. Add 50 ml of phosphoric acid-sulphuric acid mixture. Heat the flask to decompose the sample. Oxidize black residue by addition of concentrated nitric acid dropwise and heating the solution simultaneously till all carbides are decomposed and brown fumes are expelled. Dilute to 300 ml with hot water.

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

6.2 Add a few pieces of glass beads, heat the solution to boiling and add 20 ml of silver nitrate solution and 20 ml of ammonium persulphate solution adding little at a time and continue boiling till the permanganate colour develops fully (volume should be maintained at 300 ml by addition of hot water, if necessary and also boiling should be a period of 8-10 minutes). It should be ensured that sufficient persulphate is added. Wash the sides of the conical flask with water. If the colour does not develop add a few drops of potassium permanganate solution till the pink colour develops.

6.3 Add dilute hydrochloric acid dropwise to the boiling solution till permanganic acid colour is destroyed. Boil for 10 minutes more. Cool and add a known volume of standard ferrous ammonium sulphate solution until an excess of at least 5 ml is present. Titrate back with dropwise addition of standard potassium permanganate solution to a permanent pink end point which persists for 30-40 seconds.

6.4 In presence of vanadium, titrate carefully to a pink end point which persists for at least 30 to 40 seconds, to ensure complete re-oxidation of the vanadium.

6.5 The titration should be corrected for dilution effect and colour interference. The correction may be made by the following method:

6.5.1 Add same amount of ferrous ammonium sulphate as used for the sample, to the already titrated solution. Titrate with standard potassium permanganate to pink end point which lasts for 30 to 40 seconds.

7. CALCULATION

7.1 Calculate the chromium content of the steel as follows:

$$\text{Chromium, percent} = \frac{(AB - C)\,D \times 0{\cdot}017\,33 \times 100}{E}$$

where

A = volume in ml of standard ferrous ammonium sulphate solution added,

B = volume in ml of standard potassium permanganate solution equivalent to 1 ml of ferrous ammonium sulphate solution,

C = volume in ml of standard potassium permanganate solution required for titration, corrected for the blank,

D = normality of standard potassium permanganate solution, and

E = mass in g of the sample taken for the test.

7.2 Reproducibility

a) ±0·025 percent at 0·1 to 0·5 percent chromium,

b) ±0·036 percent at 0·5 to 1 percent chromium,

c) ±0·120 percent at 1 to 5 percent chromium, and

d) ±0·20 percent for chromium 5 percent and above.

APPENDIX A

(*Clause* 0.2)

INDIAN STANDARDS ON METHODS FOR CHEMICAL ANALYSIS OF STEELS

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)-1987 Determination of manganese in plain carbon and low alloy steels by arsenite method (*third revision*)

(Part 3)-1987 Determination of phosphorus by alkalimetric method (*third revision*)

(Part 4)-1987 Determination of carbon by gravimetric method (for carbon ≥ 0·1 percent) (*third revision*)

(Part 5)-1987 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel ≥ 0·1 percent) (*second revision*)

(Part 6)-1987 Determination of chromium by persulphate oxidation method (for chromium ≥ 0·1 percent) (*third 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 $\geqslant$ 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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