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Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 5 DETERMINATION OF NICKEL BY DIMETHYLGLYOXIME (GRAVIMETRIC) METHOD (FOR NICKEL ⩾ 0·1 PERCENT)

(*Third Revision*)

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METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 5 DETERMINATION OF NICKEL BY DIMETHYLGLYOXIME (GRAVIMETRIC) METHOD (FOR NICKEL ⩾ 0·1 PERCENT)

(Third Revision)

0. FOREWORD

0.1 This Indian Standard (Part 5) (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 chemial 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 5)-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 of nickel for determination from 0·5 to 0·1 percent, and

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

†Methods of chemical analysis of steels: Part 5 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel ⩾ 0·5 percent) (*second revision*).

b) inclusion of reproducibility of the method at the various levels of nickel 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 5) covers method for determination of nickel content of low alloy and high alloy steels containing more than or equal to 0·1 percent nickel.

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 complexing interfering elements, nickel in the solution of the sample is precipitated as nickel dimethylglyoximate and weighed.

5. REAGENTS

5.1 Dilute Hydrochloric Acid — 1 : 1 and 1 : 19 (*v*/*v*).

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

5.3 Perchloric Acid — 70 percent.

5.4 Tartaric Acid Solution — 50 percent (*m*/*v*).

5.5 Dilute Ammonium Hydroxide — 1 : 1 (*v*/*v*).

5.6 Dimethylglyoxime Solution — 1 percent (*m*/*v*). Dissolve 1 g of the solid reagent in 100 ml of rectified spirit.

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

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

5.7 Ammoniacal Ammonium Nitrate Solution — Dissolve 1 g of ammonium nitrate in 100 ml of water and make it slightly alkaline to methyl red with dilute ammonium hydroxide.

5.8 Acid Mixture — Make up as follows:

Concentrated hydrochloric acid (rd = 1·16 conforming to IS : 265-1976*)	400 ml
Concentrated nitric acid (rd = 1·42 conforming to IS : 264-1976†)	180 ml
Perchloric acid (rd = 1·56)	600 ml
Water	680 ml

5.9 Hydrofluoric Acid — 40 percent.

5.10 Concentrated Hydrochloric Acid — Relative density 1·16 (conforming to IS : 265-1976*).

6. PROCEDURE

6.1 For Low Alloy Steels — Weigh accurately about 2 to 3 g of the sample for nickel < 0·5 percent and 1 g for nickel up to 5 percent and transfer to a 400-ml beaker. Add 60 ml of dilute hydrochloric acid (1 : 1). Cover the beaker and digest till the decomposition is complete. Add cautiously dropwise nitric acid. Boil until iron and carbides are oxidized and brown fumes have been expelled. Add 15 to 20 ml of perchloric acid and fume, evaporate to syrupy consistency.

6.1.1 Cool and add 100 ml of water. Heat to boiling, filter to remove silica, if any, and wash with dilute hydrochloric acid (1 : 19). Cool and dilute to 250-ml in a volumetric flask.

6.1.2 Take a suitable aliquot containing about 15 to 20 mg of nickel and dilute to 200 ml. Add 10 to 15 ml of tartaric acid solution, neutralize with dilute ammonium hydroxide and add 1 ml in excess. If the solution is not clear, add more tartaric acid solution and neutralize with dilute ammonium hydroxide. Add dilute hydrochloric acid (1 : 19) until slightly acidic and warm to 60 to 80°C. Add 25 to 30 ml of dimethylglyoxime solution (too much reagent should not be used). Add dilute ammonium hydroxide until slightly alkaline (avoid addition of excess dilute ammonium hydroxide). Keep the beaker on

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

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

hot-plate at 60°C with occasional stirring for 30 minutes. Cool to room temperature.

6.1.3 Filter through previously cleaned, dried and weighed sintered glass crucible No. 3, wash the precipitate with 10 ml cold ammoniacal ammonium nitrate solution 6 to 8 times and then with the cold water. When the precipitate has been washed, discontinue the suction and dry the precipitate at 150°C to constant weight. Cool in a desiccator and weigh as nickel dimethylglyoximate.

6.2 For High Nickel and High Chromium Steels — Take 0·5 to 1 g of sample in a 400-ml beaker, add 40 ml of the acid mixture and heat gently first and when the sample has dissolved, allow it to fume and continue fuming for 7 to 10 minutes. In case the sample does not dissolve, add few drops of hydrofluoric acid and fume, and cool, dilute with 75 ml of water and filter. Wash the residue with dilute hydrochloric acid (1 : 19). Collect the filtrate and washings in a beaker, transfer to 250-ml volumetric flask and make up the volume of the filtrate to 250 ml. Complete the determination as in **6.1.2** and **6.1.3**.

6.3 For Tungsten Steels — If the steel contains tungsten, add 10 ml concentrated hydrochloric acid to solution obtained under **6.1** and dilute to 150 ml; add some ashless paper pulp, digest at 60°C and allow the precipitate to settle and filter through paper pulp pad and wash with hot dilute hydrochloric acid (1 : 19). Discard the residue. Cool the filtrate and dilute to a known volume with water. Follow further the procedure given under **6.1.1** to **6.1.3**.

NOTE — If appreciable cobalt (over 1 percent) or copper (over 1 percent) is present, dissolve the precipitate obtained under **6.1.3** into the original beaker with small quantity of alternate washes of hot dilute hydrochloric acid and warm water. Re-precipitate nickel as under **6.1.2**.

7. CALCULATION

7.1 Calculate the nickel content of the steel as follows:

$$\text{Nickel, percent} = \frac{A \times 20{\cdot}32}{B}$$

where

A = mass in g of nickel dimethylglyoximate in the aliquot, and

B = mass in g of the sample or aliquot representing the sample taken.

7.2 Reproducibility

a) ± 0·025 percent at 0·5 percent nickel and below,

b) ± 0·050 percent for nickel between 0·5 to 5 percent,

c) ± 0·120 percent for nickel between 5 to 10 percent, and

d) ± 0·160 percent for nickel above 10 percent.

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)-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 ≥ 0·5 percent) (*second revision*)

(Part 6)-1974 Determination of chromium by persulphate oxidation method (for chromium ≥ 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 ≥ 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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