भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 15 थायोसल्फेट-आयोडाइड पद्धति द्वारा ताँबा ज्ञात करना (0·05 से 5 प्रतिशत ताँबा के लिए)

(दूसरा पुनरीक्षण)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 15 DETERMINATION OF COPPER BY THIOSULPHATE IODIDE METHOD (FOR COPPER 0·05 TO 5 PERCENT)

(Second Revision)

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FOREWORD

This Indian Standard (Part 15) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 228, which was first published in 1952 and subsequently revised in 1959, 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 part covers the method for determination of copper by thiosulphate-iodide 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 2 Determination of manganese in plain carbon and low alloy steels by arsenite method

Part 3 Determination of phosphorus by alkalimetric method

Part 4 Determination of total carbon by gravimetric method (for carbon $\geqslant$ 0·1 percent)

Part 5 Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel $\geqslant$ 0·1 percent)

Part 6 Determination of chromium by persulphate oxidation method (for chromium $\geqslant$ 0·1 percent)

Part 7 Determination of molybdenum by α-benzoinoxime method (for molybdenum $\geqslant$ 0·1 percent)

Part 8 Determination of silicon by the gravimetric method (for silicon $\geqslant$ 0·1 percent)

Part 9 Determination of sulphur in plain carbon steels by evolution method

Part 10 Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum up to 0·1 percent)

Part 11 Determination of silicon by photometric 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 low and high alloy steels (for manganese 0·01 to 2·0 percent)

Part 13 Determination of arsenic

Part 14 Determination of carbon by thermal conductivity method (for carbon 0·005 to 2·000 percent)

Part 16 Determination of tungsten by spectrophotometric method (for tungsten 0·1 to 2 percent)

In this revision 'The Gravimetric Method' for determination of copper in steel has been replaced by 'The Thiosulphate Iodide Method'.

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

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 15 DETERMINATION OF COPPER BY THIOSULPHATE IODIDE METHOD (FOR COPPER 0·05 TO 5 PERCENT)

(Second Revision)

1 SCOPE

This standard (Part 15) describes the thiosulphate-iodide method for determination of copper in steel in range from 0·05 to 5 percent.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)

3 SAMPLING

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

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) shall be employed for the test.

5 DETERMINATION OF COPPER

5.1 Outline of the Method

The sample is dissolved in dilute sulphuric acid and copper is precipitated with sodium-thiosulphate. Precipitate is ignited, dissolved in acid and determined iodometrically.

5.2 Reagents

5.2.1 *Dilute Sulphuric Acid*, 1 : 9 (*v/v*)·

5.2.2 *Sodium Thiosulphate Solution*

Dissolve 100 g of sodium thiosulphate ($Na_2S_2O_3$ $5H_2O$) in 100 ml of water and filter if a hazy solution is obtained.

5.2.3 *Wash Solution*

Saturate dilute sulphuric acid solution 1 : 99 (*v/v*) with hydrogen sulphide.

5.2.4 *Dilute Nitric Acid*, 60 percent.

NOTE — Nitric acid conforming to IS 264 : 1976 contains 70 percent of nitric acid.

5.2.5 *Sodium Fluoride*, solid.

5.2.6 *Dilute Ammonium Hydroxide*, 1 : 1 (*v/v*).

5.2.7 *Acetic Acid*, 80 percent.

5.2.8 *Potassium Iodide*, solid.

5.2.9 *Starch Solution*, 0·5 percent.

Make a suspension of 0·5 g of starch in 10 ml of water. Add to it 90 ml of boiling water, cool and mix.

5.2.10 *Standard Sodium Thiosulphate Solution (N/50)*

Dissolve 5 g of sodium thiosulphate ($Na_2S_2O_3$. $5H_2O$) in 500 ml of water, add 0·1 g of sodium carbonate and dilute to 1 litre. Standardize against pure copper.

5.2.10.1 *Standardization*

Transfer 0·05 g of pure copper to 150 ml beaker, cover and dissolve in 4-5 ml of dilute nitric acid (3 : 5). Boil gently to expel oxides of nitrogen. Cool and add ammonium hydroxide (1 : 1) until the solution just turns blue. Add 5 ml of acetic acid and then 1 ml in excess. Complete the titration as described in **5.3.2** to **5.3.4**. Find out the copper equivalent for 1 ml of thiosulphate solution.

5.2.11 *Ammonium Bifluoride Solution*, 200 g/l.

5.3 Procedure

5.3.1 *Test Portion*

Weigh a sample nearest to 1 mg as follows:

Copper, Percent	*Mass in g of Sample*
Up to 0·25	5
0·25 to 1	2
1 to 1·5	1
1·5 to 5	0·5

5.3.2 Weigh the quantity of sample as per copper content mentioned above and transfer to 500 ml beaker. Add 100 ml of dilute sulphuric acid (*see* Note). Heat gently until reaction ceases, dilute to 250 ml. Heat to boil, add 10 ml of sodium thiosulphate solution in small portions and continue to boil for 5-10 minutes or until the precipitate settle rapidly. Filter, and wash the precipitate with wash solution. Place the paper and precipitate in a porcelain or silica crucible, dry and ignite at a low temperature (520 to 550°C) until all carbon is destroyed. Cool, and transfer the contents of the crucible to 250 ml beaker. Add 5-6 ml of dilute nitric acid to the crucible, warm gently and pour upon the contents in the beaker. Rinse the crucible with water and warm the beaker and the contents until the copper oxide has dissolved.

NOTE — For samples not dissolving in dilute sulphuric acid, use mixture of hydrochloric acid, nitric acid and 10 ml of sulphuric acid. Heat to fumes, cool and repeat the operation once more.

5.3.3 Carefully, evaporate the solution to 2-3 ml. Cool, add 30 ml of water and either 5 ml of ammonium bifluoride solution or 1 g of sodium fluoride. Add ammonium hydroxide until the solution is just alkaline as noticed by the blue colour. Cool the solution to room temperature.

5.3.4 Acidify the solution with acetic acid and add 1 ml in excess. Add 3-4 g of potassium iodide and stir well. Immediately titrate with standard sodium thiosulphate solution. When the brown tint has nearly disappeared, add 5 ml of starch solution and continue titration until with one drop it changes the colour from blue to yellowish white and remains permanent for 15 to 20 seconds (*see* Notes).

NOTES

1 Add 5 ml of potassium thiosulphate solution (10 percent) to get a better end point detection.

2 If coper is present in small amount, estimate copper by diethyldithio-carbamate spectrophotometric method.

5.3.5 *Blank*

Carry out a blank determination following the procedure specified in **5.3.2** to **5.3.4** using same amount of all reagents but without the sample.

5.3.6 *Calculation*

$$\text{Copper, percent by mass} = \frac{(A - B) \times C \times 100}{D}$$

where

A = volume in ml, of $Na_2S_2O_3$ solution required for the sample;

B = volume in ml, of $Na_2S_2O_3$ solution required for the blank;

C = copper equivalent (in g/ml) of $Na_2S_2O_3$ solution; and

D = mass in g, of sample taken.

5.4 Reproducibility

±0·01 percent at 0·1 percent copper level.

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Amendments Issued Since Publication

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