IS 12614-6 (1988): Methods of Chemical Analysis of Ferro-molybdenum, Part 6: Determination of Copper [MTD 5: Ferro Alloys]

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IS 12614 (Part 6) : 1988

(Reaffirmed 1999)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF FERRO-MOLYBDENUM

PART 6 DETERMINATION OF COPPER

भारतीय मानक

फैरो-मालिबडेनम के रासायनिक विश्लेषण की पद्धति

भाग 6 तांबा ज्ञात करना

UDC 669·15'28-198 : 543 [546·56]

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

September 1989

Price Group 1

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, SMDC 2

FOREWORD

This Indian Standard (Part 6) was adopted by the Bureau of Indian Standards on 17 October 1988, 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.

IS 1559 : 1961 'Methods of chemical analysis of ferro-alloys' covers the chemical analysis of different ferro-alloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silico-manganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten. While reviewing this standard, the sectional committee had decided that a separate series for each ferro-alloy be prepared. Accordingly IS 1559 in its various parts was published for the chemical analysis of ferrosilicon.

The chemical analysis of ferromolybdenum is covered into series of parts of this standard. With the publication of these parts, the analysis of the relevant constituents prescribed for ferromolybdenum in IS 1559 : 1961 will be superseded. This part covers the determination of copper. The other parts are:

Part 1 Determination of molybdenum
Part 2 Determination of total carbon
Part 3 Determination of silicon
Part 4 Determination of sulphur
Part 5 Determination of phosphorus
Part 7 Determination of aluminium

In this revision, the method for determination of copper by thiosulphate iodide and spectrophotometric methods have been incorporated.

In reporting the result of 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 OF CHEMICAL ANALYSIS OF FERRO-MOLYBDENUM

PART 6 DETERMINATION OF COPPER

1 SCOPE

This standard (Part 6) covers the volumetric and spectrophotometric method for determination of copper in ferromolybdenum.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard :

IS No.	*Title*
IS 264 : 1976	Specification for nitric acid (*second revision*)
IS 265 : 1976	Specification for hydrochloric acid (*second revision*)
IS 266 : 1977	Specification for sulphuric acid (*second revision*)
IS 1472 : 1977	Methods of sampling ferro-alloys for determination of chemical composition (*first revision*)

3 SAMPLING

Samples shall be drawn and prepared in accordance with IS 1472 : 1977.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the tests.

5 DETERMINATION OF COPPER IN FERRO-MOLYBDENUM BY THIOSULPHATE IODIDE METHOD

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 Nitric Acid,* 1 : 3 (*v*/*v*).

5.2.2 *Hydrofluoric Acid,* 40 percent.

5.2.3 *Concentrated Sulphuric Acid,* rd = 1·84 (conforming to IS 266 : 1977).

5.2.4 *Sodium Hydroxide Solution,* 10 percent (*m*/*v*).

5.2.5 *Dilute Sulphuric Acid,* 1 : 3, 1 : 4 and 1 : 99 (*v*/*v*).

5.2.6 *Sodium Thiosulphate Solution,* 50 percent (*m*/*v*).

5.2.7 *Hydrogen Sulphide,* gas.

5.2.8 *Concentrated Nitric Acid,* rd = 1·42 (conforming to IS 264 : 1976).

5.2.9 *Sodium Fluoride,* solid.

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

5.2.11 *Dilute Acetic Acid Solution,* 80 percent (*m*/*v*).

5.2.12 *Potassium Iodide,* solid.

5.2.13 *Standard Sodium Thiosulphate Solution*

Dissolve 5 g of sodium thiosulphate ($Na_2S_2O_3.3H_2O$) in 500 ml of water, add 0·1 g of sodium carbonate and dilute to 1 litre. Standardize against pure copper.

5.2.13.1 *Standardization*

Transfer 0·05 g of pure copper to 150 ml beaker, dissolve in 4 to 5 ml of dilute nitric acid (1 : 1). Boil gently to expel oxide of nitrogen, cool, add ammonium hydroxide (1 : 1) until solution just turns blue. Add aceticacid (5 to 10 ml). Complete the titration as described in **5.3**.

5.3 Procedure

5.3.1 Transfer 2·00 g of sample to 400 ml beaker and dissolve in 50 ml dilute nitric acid. Add 2 to 3 drops of hydrofluoric acid. Add 5 ml of concentrated sulphuric acid and evaporate to fumes. Cool and add 150 ml of water. Filter the solution through Whatman filter paper No. 541, and wash the residue with hot water.

5.3.2 Neutralize the filtrate with sodium hydroxide solution. Add 100 ml of warm solution of sodium hydroxide. Filter and discard the filtrate. Dissolve the precipitate in dilute sulphuric acid (1 : 3) and repeat the precipitation and filtration.

5.3.3 Dissolve the precipitate with 50 ml of dilute sulphuric acid (1 : 4) in the beaker in which precipitation was carried. Dilute to 300 ml, add 5 ml of sodium thiosulphate solution (50 percent) and boil for 5 minutes. Add 50 ml more sodium thiosulphate solution (50 percent) and boil for 15 minutes.

5.3.4 Filter the precipitate, wash with dilute sulphuric acid (1 : 99) (saturated with hydrogen sulphide). Place the paper and precipitate in a porcelain or silica crucible and ignite at a temperature of 520-550°C. Cool and transfer the contents of crucible to 250 ml beaker. Add 5 to 6 ml of concentrated nitric acid. Evaporate the solution to oxides of nitorgen, cool, add 90 ml of water and 1 g of sodium fluoride. Add dilute ammonium hydroxide until alkaline to litmus. Neutralize the solution with dilute acetic acid and add 1 ml in excess. Add 3 to 4 g of potassium iodide (dissolved in little water) and titrate with standard sodium thiosulphate solution (0·02 N) when the brown tints have nearly disappeared. Add 5 ml of starch solution and continue titration until yellowish white colour is obtained for 15 to 20 seconds.

5.3.5 *Blank*

Carry out the reagent blank using the same quantity of reagents.

5.3.6 *Calculation*

Copper, percent by mass

$$= \frac{(A - B) \times C \times 100}{D}$$

where

A = volume of standard sodium thiosulphate solution (0·02 N) required for test solution,

B = volume of standard sodium thiosulphate (0·02 N) required for the blank,

C = copper equivalent in g/ml of standard sodium thiosulphate solution, and

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

6 DETERMINATION OF COPPER IN FERRO MOLYBDENUM BY SPECTROPHOTOMETRIC (DIETHYL DITHIOCARBAMATE METHOD)

6.1 Outline of the Method

Copper is complexed with diethyl dithiocarbamate and extracted with carbon tetrachloride solution and measured at 430 nm.

6.2 Reagents

6.2.1 *Mix Acids*

Mix hydrochloric acid, nitric acid and sulphuric acid in the ratio of 1 : 3 : 3.

6.2.2 *Citric Acid,* 20 percent.

6.2.3 *Concentrated Ammonia Solution,* rd = 0·90.

6.2.4 *EDTA Solution,* 1 percent (*m/v*).

6.2.5 *Sodium Diethyl Dithiocarbamate Solution,* 0·1 percent (*m/v*).

6.2.6 *Carbon Tetrachloride*

6.2.7 *Standard Copper Solution*

Dissolve 0·100 g of electrolytic copper in 5 ml of dilute nitric acid (1 : 1), evaporate to near dryness and dilute to 100 ml in a volumetric flask. Take 10 ml of the solution and dilute to 100 ml (1 ml = μg Cu)

6.3 Procedure

6.3.1 Weigh 0·100 g of sample into 100 ml beaker. Add 10 ml of mix acids and heat to remove nitrous oxide fumes. Cool, transfer the solution to 100 ml volumetric flask and make up.

6.3.2 Take 10 ml aliquot of the solution into 100 ml of separating funnel, add 10 ml of citric acid solution and mix. Add 5 ml of ammonia solution and mix by swirling (*p*H of the solution should be 8·5 to 9·0). Add 2 ml of EDTA solution. Add 10 ml of sodium diethyl dithiocarbamate solution and mix by shaking. Add 10 ml of carbon tetrachloride (CCl_4) solution and shake for 3 minutes. Filter the organic phase (CCl_4) solution through dry filter paper and measure the absorbance of complex at 430 nm against blank solution of carbon tetrachloride.

6.3.3 *Calibration Curve*

Take 0, 1, 2, 3, 4 and 5 ml of standard copper solution (0 to 50 μg Cu) into six 100 ml separating funnels. Add 10 ml of water and proceed according to **6.3.2.** Plot the calibration curve of the absorbance values of standard solution against mg of copper.

6.3.4 *Calculation*

Convert the absorbance values of test solution to mg of copper by means of calibration curve, and calculate the percentage of copper as follows :

$$\text{Copper, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg of copper found in the aliquot solution, and

B = mass in g of sample represented by aliquot taken.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75
Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices:	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	331 01 31 331 13 75
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	36 24 99
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	2 18 43 3 16 41
Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113	41 24 42 41 25 19 41 29 16
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