भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 16 स्पेक्ट्रम प्रकाशमिति पद्धति द्वारा टँग्स्टन ज्ञात करना (0·1 से 2 प्रतिशत टँग्स्टन के लिए)

(दूसरा पुनरीक्षण)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 16 DETERMINATION OF TUNGSTEN BY SPECTROPHOTOMETRIC METHOD (FOR TUNGSTEN 0·1 TO 2 PERCENT)

(Second Revision)

First Reprint SEPTEMBER 1996

UDC 669·14 : 543·42 [546·78]

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

September 1992

Price Group 1

FOREWORD

This Indian Standard (Part 16) (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, alongwith 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 tungsten. The other parts of this series are:

IS 228 Methods for chemical analysis of steels:

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 15 Determination of copper by thiosulphate iodide method (for copper 0·05 to 5 percent)

In this revision the spectrophotometric method for determination of tungsten in steel has been prescribed. The reproducibility of the method has been given on the basis of interlaboratory testing.

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 (*rivised*)'.

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 16 DETERMINATION OF TUNGSTEN BY SPECTROPHOTOMETRIC METHOD (FOR TUNGSTEN 0·1 TO 2 PERCENT)

(Second Revision)

1 SCOPE

This standard (Part 16) describes the spectrophotometric method for determination of tungsten in the range 0·1 to 2 percent in alloy steels.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard.

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1987	Hydrochloric acid (*third 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 TUNGSTEN

5.1 Outline of the Method

In presence of potassium thiocynate, tungsten in hydrochloric acid is reduced with titanium chloride and stannous chloride to form yellowish-green tungsten thiocynate complex and measured at 400 nm.

5.2 Reagents

5.2.1 *Acid Mixture*

Mix 150 ml of sulphuric acid (r. d = 1·84) and 150 ml of phosphoric acid and make up to 1 litre with water.

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

5.2.3 *Sodium Hydroxide Solution*, 16 and 30 percent (*m/v*).

5.2.4 *Hydrochloric Acid*, rd = 1·16 (conforming to IS 266 : 1987).

5.2.5 *Potassium Thiocynate Solution*, 50 percent (*m/v*).

5.2.6 *Stannous Chloride Solution*

Dissolve 36 g of stannous chloride in 20 ml of dilute hydrochloric acid (1 : 1) and dilute to 100 ml. Prepare fresh as and when required.

5.2.7 *Titanium Trichloride Solution*, 15 percent (*m/v*).

Take 15·0 g of titanium metal, add 80 ml of hydrochloric acid and reflux. Finally make up to 100 ml with hydrochloric acid.

5.2.8 *Standard Tungsten Solution* (1 ml = 0·10 mg)

Dissolve 0·1794 g of sodium tungsten dihydrate ($Na_2WO_4 . 2H_2O$) in 50 ml of water. Transfer to a 100-ml volumetric flask, dilute to volume, and mix. Transfer 10 ml aliquot of this solution to a 100-ml volumetric flask, dilute to volume, and mix.

5.3 Procedure

5.3.1 Transfer 0·5 g of sample to a 200-ml beaker. Add 30 ml of acid mixture and oxidize with few drops of nitric acid. Heat to fumes, cool and add 20 ml of water. Add this solution drop by drop to sodium hydroxide (30 percent) till neutralization. Transfer the solution into 250-ml volumetric flask containing 50 ml hot sodium hydroxide solution (16 percent) shaking the volumetric flask throughout the transfer. Cool and make up to the mark. Shake well and allow to settle.

5.3.2 Filter the solution through medium textured filter paper. Take 20 ml aliquot of the filtrate into 100-ml volumetric flask. Add 30 ml water, 20 ml hydrochloric acid, 5 ml of potassium thiocynate, 1 ml of stannous chloride solution and 0·3 ml of titanium chloride solution (*see* Note).

Make up to volume with hydrochloric acid and shake vigorously. Allow to stand for 30 minutes and measure the absorbance against reagent blank at 400 nm.

NOTE — While adding each of the above reagents to the filtrate shake the flask in each addition.

5.3.3 *Blank*

Carry out a blank determination using 0·5 g of pure iron and following the procedure specified in **5.3.1** and **5.3.2**, using same amount of all reagents but without the sample.

5.3.4 *Calibration Solution*

Take 0, 2, 4, 6, 8, 10 and 12 ml of standard tungsten solution *see* (**5.2.8**) into seven 100-ml volumetric flasks. Add the same quantity of reagents mentioned in **5.3.2** and measure the absorbance values of standard solutions against the first solution. Plot absorbance values against mg of tungsten per 100 ml of solution. Compute the tungsten content of the sample from the calibration curve.

5.4 Calculation

Calculate the percentage of tungsten as follows:

$$\text{Tungsten, percent by mass} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg, of tungsten found in 100 ml of final solution, and

B = mass in g, of sample represented by 100 ml of final solution

5.5 Reproducibility

± 0·02 at 0·5 percent level,

± 0·03 at 1 percent level and,

± 0·05 at 2 percent level.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

This Indian Standard has been developed from Doc: No. MTD 2 (3357)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 323 01 31, 323 33 75, 323 94 02

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices:		Telephone
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	323 76 17, 323 38 41
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola CALCUTTA 700054	337 84 99, 337 85 61 337 86 26, 337 91 20
Northern	: SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43 60 20 25
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 02 16, 235 04 42 235 15 19, 235 23 15
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	832 92 95, 832 78 58 832 78 91, 832 78 92
Branches	: AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. PATNA. PUNE. THIRUVANANTHAPURAM.	

Printed by Reprography Unit, BIS, New Delhi