IS 228 (Part 10) : 1989
(Reaffirmed 1994)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 10 DETERMINATION OF MOLYBDENUM BY THIOCYANATE (PHOTOMETRIC) METHOD IN LOW AND HIGH ALLOY STEELS (FOR MOLYBDENUM 0·01 TO 1·50 PERCENT)

(Third Revision)

भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 10 अल्प और उच्च मिश्र इस्पातों में (0·01 से 1·50) प्रतिशत मॉलीब्डनम के लिए थायोसाईनेट (प्रकाशमिति) पद्धति द्वारा मॉलीब्डनम ज्ञात करना

(तोसरा पुनरीक्षण)

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FOREWORD

This Indian Standard (Part 10) (Third Revision) was adopted by the Bureau of Indian Standards on 22 December 1989, 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. This standard was again revised to make it comprehensive in respect of steel analysis and to exclude pig iron and cast iron which are being covered in separate standards. 14 parts have already been issued covering only chemical analysis of steels.

This standard IS 228 (Part 10) was published in 1976. In this revision the wave length at which the intensity of the molybdenum complex is measured has been modified and the reproducibility of the method has been incorporated.

In reporting the results 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

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1 SCOPE

1.1 This standard (Part 10) covers the method for the determination of molybdenum in the range 0·01 to 1·50 percent by thiocyanate (photometric) method in low and high alloy steels.

2 REFERENCE

2.1 The following Indian Standard is a necessary adjunct to this standard:

IS No.	*Title*
265 : 1987	Specification for hydrochloric acid (*third revision*)

3 SAMPLING

3.1 Samples shall be drawn and prepared as prescribed in the relevant Indian Standard.

4 QUALITY OF REAGENTS

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

5 DETERMINATION OF MOLYBDENUM BY THIOCYANATE (PHOTOMETRIC) METHOD

5.1 Outline of the Method

Sample aliquot is treated with thiocyanate to develop molybdenum and iron complexes and reduced with stannous chloride. Molybdenum complex is extracted with butyl acetate and measured at 470 nm.

5.2 Reagents

5.2.1 *Acid Mixture*

To 700 ml of perchloric acid (rd = 1·67) add 150 ml of phosphoric acid (rd = 1·70) and 150 ml of nitric acid (rd = 1·42). Mix thoroughly while adding the acids.

5.2.2 *Concentrated Hydrochloric Acid,* rd = 1·16 (conforming to IS 265 : 1987).

5.2.3 *Dilute Sulphuric Acid,* 1 : 1 and 1 : 5 (*v/v*).

5.2.4 *Potassium Thiocyanate Solution,* 100 g/litre in water.

5.2.5 *Stannous Chloride Solution (100 g/l)*

Dissolve 20 g of stannous chloride dihydrate ($SnCl_2$, $2H_2O$) in 25 ml concentrated hydrochloric acid (rd = 1·16) with gentle stirring. Heat to 60 to 70°C to dissolve completely. Cool and dilute to 200 ml with water. Add a few pieces of metallic tin and store in a stoppered bottle.

5.2.6 *N-butyl Acetate*

5.2.7 *Iron-Molybdenum Free (Mo < 0·001 percent)*

5.2.8 *Standard Molybdenum Solution (1 ml = 0·01 mg Mo)*

Transfer 0·100 0 g of molybdenum metal (9·99 percent pure) to a 250 ml conical flask and dissolve in 10 ml concentrated hydrochloric acid and 10 ml water. Add 1 ml of concentrated nitric acid and heat to dissolution. Cool, dilute to 1 litre in a volumetric flask. Take 100 ml of this solution and dilute to 1 litre with water.

5.3 Procedure

5.3.1 Transfer 0·500 g of the sample to a 250 ml conical flask. Add 25 ml of the acid mixture. Heat gently to dissolve the sample. Evaporate the solution till fumes of perchloric acid evolves and continue heating further till all the chromium, if present, is oxidised. Add dropwise concentrated hydrochloric acid till most of the chromium is volatilised. Cool, add 50 ml of dilute sulphuric acid (1 : 1), heat to boiling (to expel any free chlorine) and cool. Transfer the solution to a 100 ml volumetric flask and dilute to mark with water. Mix thoroughly.

5.3.2 Pipette out a suitable aliquot containing 0·05 to 0·1 mg molybdenum into a 250 ml separating funnel. Add 15 ml of potassium thiocyanate solution and 15 ml of stannous chloride solution. Mix thoroughly after each

addition. Add 25 ml of butyl acetate, stopper and shake vigorously for one minute. Allow the phases to separate. Remove the stopper and drain out the aqueous phase. Add to the organic layer 50 ml of dilute sulphuric acid (1 : 5), 5 ml of potassium thiocyanate and 5 ml of stannous chloride solution. Stopper and shake vigorously for one minute. Allow the phases to separate and drain off the aqueous layer and discard.

5.3.3 Collect the organic phase in a funnel after passing through a dry filter paper. Transfer a suitable portion of butyl acetate as reference solution to a 1 cm cell and measure the absorbance of test soluion against it at 470 nm.

5.3.4 *Blank*

Carry out a blank determination using all the reagents and 0·5 g iron instead of the sample. Record the absorbance of the blank solution using butyl acetate as reference solution.

5.3.5 *Calibration Curve*

Take 0·500 0 g of iron in each of seven 250 ml conical flasks. Transfer 0, 1, 2, 4, 6, 8 and 10 ml of standard molybdenum solution (1 ml = 0·01 mg Mo) to each of these. Add 25 ml of the acid mixture and proceed as specified in **5.3.1** to **5.3.3**. The flask containing zero ml of Mo is taken as blank and the absorbance values are plotted against milligram of molybdenum.

5.3.6 *Calculation*

Deduct the absorbance value of blank from the test solution and the calibration curve. Find out the mg of molybdenum present in the aliquot portion of the test solution taken. Calculate the percentage of molybdenum as follows:

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

where

A = mass in mg of Mo in the aliquot portion of the test solution; and

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

5.3.7 *Reproducibility*

± 0·005 at 0·05 percent level

± 0·02 at 0·15 percent level

± 0·04 at 0·5 percent level

± 0·06 at 1 percent level

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