IS 228 (Part 7) : 1990
(Reaffirmed 1996)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 7 DETERMINATION OF MOLYBDENUM BY ALPHA-BENZOINOXIME METHOD IN ALLOY STEELS (FOR MOLYBDENUM > 1 PERCENT AND NOT CONTAINING TUNGSTEN)

(Third Revision)

भारतीय मानक

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 7 एल्फा बेन्जाइनआक्साइम् पद्धति द्वारा मालीब्डनम् ज्ञात करना
(मालीब्डनम् के लिए > 1 प्रतिशत और टंगस्टन रहित)

(तीसरा पुनरीक्षण)

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BUREAU OF INDIAN STANDARDS
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Price Group 1

FOREWORD

This Indian Standard (Part 7) (Third Revision) was adopted by the Bureau of Indian Standards on 23 February 1990, 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 are being covered in separate standards. During its second revision, the standard was split up in several parts and 14 parts have already been published covering only chemical analysis of steels.

This standard IS 228 (Part 7) was published in 1974. In this third revision, the part has been updated.

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 7) covers the alpha-benzoinoxime method for determination of molybdenum content in low alloy and high alloy steels containing molybdenum above 1 percent and no tungsten.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard.

IS No.	*Title*
264 : 1976	Specification for nitric acid (*second revision*)
265 : 1987	Specification for hydrochloric acid (*third revision*)

3 SAMPLING

3.1 The samples shall be drawn and prepared as prescribed in the relevant Indian Standards.

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 ALPHA-BENZOINOXIME METHOD

5.1 Outline of the Method

Molybdenum is preciptated with alpha-benzoinoxime, and the precipitate is ignited at 500-525°C and weighed as MoO_3.

5.2 Reagents

5.2.1 *Dilute Sulphuric Acid,* 1 : 1, 1 : 4 and 1 : 6 (*v/v*).

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

5.2.3 *Hydroflouric Acid,* 40 percent.

5.2.4 *Boric Acid Solution,* 4 percent.

5.2.5 *Potassium Bisulphite,* Solid.

5.2.6 *Ferrous Sulphate Solution,* 5 percent.

Dissolve 5 g of ferrous sulphate in water containing 5 ml of sulphuric acid and dilute to 100 ml.

5.2.7 *Alpha-Benzoinoxime Solution,* 2 percent

Dissolve 2 g of alpha-benzoinoxime in 100 ml of ethanol. Filter if the solution is not clear.

5.2.8 *Bromine Water*

Saturate 100 ml of water with bromine, adding 1 to 2 ml of bromine at a time till few drops of it remain undissolved.

5.2.9 *Sulphuric Acid-Benzoinoxime Wash Solution*

To 1 litre of dilute sulphuric acid (1 : 99), add 5 ml of alpha-benzoinoxime solution (2 percent).

5.2.10 *Dilute Ammonium Hydroxide Solution,* 1 : 1 and 1 : 99 (*v/v*).

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

5.2.12 *Dilute Hydrochloric Acid,* 1 : 1 and 1 : 50 (*v/v*).

5.2.13 *Tartaric Acid,* solid.

5.2.14 *Hydrogen Sulphide,* gas.

5.2.15 *Hydrogen Sulphide Wash Solution*

Saturate dilute sulphuric acid (1 : 99) with hydrogen sulphide.

5.2.16 *Cinchonine Solution*

Dissolve 10 g cinchonine hydrochloride in 100 ml of dilute hydrochloric acid (1 : 1).

5.2.17 *Cinchonine Wash Solution*

Dilute 30 ml of cinchonine solution to 1 litre with water.

5.3 Procedure

5.3.1 Dissolve 1·00 to 3·00 g of sample in 50 ml dilute sulphuric acid (1 : 6) in a 400-ml beaker and warm till the reaction ceases. Add drop by drop concentrated nitric acid to decompose carbides and to oxidise iron and molybdenum. Boil to expel the nitrous fumes. Add 2 to 4 drops of hydrofluoric acid and allow to react. Add 10 ml of boric acid solution. Boil for a few minutes. Dilute to 100 ml. Filter and wash with hot dilute hydrochloric acid (1 : 99). Ignite the filter paper at low temperature (below 500°C), fuse with potassium bisulphite and dissolve in water. Add to the filtrate.

5.3.2 Dilute to 100 ml. Cool to about 10°C. Add 10 ml of ferrous sulphate solution. Add a few pieces of ashless paper pulp and stir, and add slowly 10 ml of alpha-benzoinoxime solution with constant stirring. (Add 5 ml more for each 0·01 g of molybdenum, followed by the addition of sufficient bromine water to impart a pale yellow colour to the solution and finally 3 to 4 ml more of the reagent). Allow the solution to remain in the cooling mixture for 10 minutes, while stirring occasionally. Filter through a medium textured ashless filter paper. If the filtrate is not clear, filter through the same filter paper. Wash six to seven times with cold sulphuric acid-benzoinoxime solution.

5.3.3 Transfer the precipitate and paper to a weighed platinum crucible, dry and ignite at 500-525°C to constant mass and weigh (A). Dissolve the oxide in 5 to 10 ml of dilute ammonium hydroxide solution (1 : 1), digest and wash the residue with hot dilute ammonium hydroxide solution (1 : 99). Ignite the residue in the same platinum crucible and weigh (B). The difference of weight (A — B) represents the weight of MoO_3.

5.3.4 If the ammonical filtrate is blue in colour, indicating the presence of copper; estimate copper by diethyl dithiocarbamate — spectrophotometric method.

5.3.5 *For Tungsten Steel*

5.3.5.1 Take 1·00 g of the sample and add 10 ml of concentrated hydrochloric acid. Heat till reaction subsides. Add concentrated nitric acid in small quantity and digest till bright yellow precipitate of tungstic oxide is formed. Evaporate to syrupy consistency. Repeat the evaporation once more with 5 ml of concentrated hydrochloric acid. Add 5 ml of concentrated hydrochloric acid and dilute to 100 ml. Boil for one or two minutes and allow to settle for 15 minutes. Filter through a paper pad and wash the residue thoroughly with dilute hydrochloric acid (1 : 50) and twice with hot water. Reserve the filtrate and washings (F_1).

5.3.5.2 Digest the precipitate (**5.3.5.1**) with 30 ml of dilute ammonium hydroxide solution (1 : 1), filter and wash with dilute ammonium hydroxide (1 : 99). Add 5 g of tartaric acid to the filtrate. Neutralize the solution with dilute hydrochloric acid (1 : 1) and add about 10 ml of concentrated hydrochloric acid. Dilute to 100 ml and warm. Pass the hydrogen sulphide gas under pressure. Filter and wash with hydrogen sulphide wash solution. Discard the filtrate. Ignite the residue at 500-525°C. Cool and dissolve in 20 ml of dilute ammonium hydroxide solution (1 : 1) and filter. Mix the filtrate with the filtrate F_1 obtained in **5.3.5.1**. Dilute the combined filtrate to 200 m and follow the procedure as specified in **5.3.2** and **5.3.3**.

5.3.6 *For High Silicon Steel*

5.3.6.1 Take 1·00 g of the sample, add 10 ml of concentrated hydrochloric acid and a few drops of concentrated nitric acid dropwise. Evaporate to syrupy consistency. To further dehydrate, add 5 ml of concentrated hydrochloric acid, evaporate and bake. Cool and add 5 ml of concentrated hydrochloric acid and dilute to 100 ml. Boil for one or two minutes and allow to settle for 15 minutes. Filter through a filter pad and wash the residue thoroughly with dilute hydrochloric acid (1 : 50) and then twice with shot water. Reserve the filtrate and washing (F_2).

5.3.6.2 Ignite the residue in a platinum crucible at a temperature 500-525°C. Cool and add 2 ml of dilute sulphuric acid (1 : 4) and 2 ml of hydrofluoric acid. Evaporate to fumes and add another 2 ml of hydrofluoric acid and evaporate again. Cool, dilute with water, warm again and filter through filter pad. Add this filtrate to the filtrate F_2 obtained in **5.3.6.1**. Dilute the combined filtrate to 200 ml and follow the procedure as specified in **5.3.2** and **5.3.3**.

6 CALCULATION

6.1 Calculate the molybdenum content as follows:

$$\text{Molybdenum, percent by mass} = \frac{(A - B) \times 66{\cdot}7}{C}$$

where

$A - B$ = mass, in g, of molybdenum oxide obtained under **5.3.3**, and

C = mass, in g, of the sample taken for the test.

Bureau of Indian Standards

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Review of Indian Standards

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