

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ
भाग 7 अल्फा बेन्ज़ाइनोक्सिम पद्धति द्वारा
मॉलिब्डेनम ज्ञात करना (मॉलिब्डेनम के
लिए > 1 प्रतिशत और टंगस्टन रहित)
(चौथा पुनरीक्षण)

**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)**

(Fourth Revision)

ICS 77.080.20

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FOREWORD

This Indian Standard (Part 7) (Fourth Revision) was adopted by the Bureau of Indian Standards on recommendation of the Methods of Chemical analysis of Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1952 and subsequently revised in 1959, 1974 and 1990. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

This part covers method for the determination of molybdenum by alpha-benzoinoxime method in alloy steels (for molybdenum > 1 percent and not containing tungsten). 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 \geq 0.1 percent)
- Part 5 Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel \geq 0.1 percent)
- Part 6 Determination of chromium by persulphate oxidation method (for chromium \geq 0.1 percent)
- Part 8 Determination of silicon by gravimetric method (for silicon 0.05 to 5.00 percent)
- Part 9 Determination of sulphur by evolution method (for sulphur 0.01 to 0.25 percent)
- Part 10 Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum 0.01 to 1.5 percent)
- Part 11 Determination of total silicon by reduced molybdosilicate spectrophotometric 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 plain carbon, low alloy and high alloy steels (for manganese 0.01 to 5.0 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)
- Part 16 Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent)
- Part 17 Determination of nitrogen by thermal conductivity method (for nitrogen up to 0.04 percent)
- Part 18 Determination of oxygen by instrumental method (for oxygen 0.001 to 0.100 0 percent)
- Part 19 Determination of nitrogen by steam distillation method (for nitrogen 0.002 to 0.50 percent)
- Part 20 Determination of total carbon and sulfur content — Infrared absorption method after combustion in an induction furnace (routine method)
- Part 21 Determination of copper by spectrometric method (for copper 0.02 to 0.5 percent)
- Part 22 Determination of total hydrogen in steel by thermal conductivity method (hydrogen 0.1 ppm to 50 ppm)
- Part 23 Determination of total nitrogen in steel by optical emission spectrometer (nitrogen 0.002 to 1.0 percent)

(Continued on third cover)

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)

(*Fourth Revision*)

1 SCOPE

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

The standards given below contain provisions, which through reference in this text, constitute provision of this standard. At the time of the publication, the editions indicated below were valid. All the standards are subject to revision, and parties to agreement based on these standards are encouraged to investigate the possibility of applying the most recent edition of these standards:

| <i>IS No.</i> | <i>Title</i> |
|---------------|--|
| IS 264 : 2005 | Nitric acid — Specification (<i>third revision</i>) |
| IS 265 : 2021 | Hydrochloric acid — Specification (<i>fifth revision</i>) |

3 SAMPLING

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

4 QUALITY OF REAGENTS

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 precipitated with alpha-benzoinoxime, and the precipitate is ignited at 500 °C to 525 °C and weighed as MoO₃.

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)

5.2.3 Hydrofluoric 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 the solution is not clear.

5.2.8 Bromine Water

Saturate 100 ml of water with bromine, adding 1 ml to 2 ml of bromine at a time till few drops of it remains 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)

5.2.12 Dilute Hydrochloric Acid — 1 : 1 and 1 : 50 (v/v)

5.2.13 Tartaric Acid — solid

To access Indian Standards click on the link below:

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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 g 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 oxidize 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 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 ml to 4 ml more of the reagent). Allow the solution to remain in the cooling mixture for 10 min, 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 °C to 525 °C to constant mass and weigh (A). Dissolve the oxide in 5 ml 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 MoO₃.

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 min. 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 (F₁).

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 °C to 525 °C. Cool and dissolve in 20 ml of dilute ammonium hydroxide solution (1 : 1) and filter. Mix the filtrate with the filtrate F₁ obtained in **5.3.5.1**. Dilute the combined filtrate to 200 ml 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 min. 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 (F₂).

5.3.6.2 Ignite the residue in a platinum crucible at a temperature 500 °C to 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 F₂ 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:

$$\text{Molybdenum, percent by mass} = \frac{(A - B) \times 66.7}{C}$$

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.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

| <i>Organization</i> | <i>Representative(s)</i> |
|---|---|
| CSIR - National Metallurgical Laboratory, Jamshedpur | DR SANCHITA CHAKRAVARTY (Chairperson) |
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| Bhabha Atomic Research Centre, Mumbai | MS SANJUKTA A. KUMAR SHRI M. V. RANA (<i>Alternate</i>) |
| CSIR - National Metallurgical Laboratory, Jamshedpur | DR ASHOK K. MOHANTY (<i>Alternate</i>) |
| Defence Metallurgical Research Laboratory, Ministry of Defence, Hyderabad | SHRI S. S. KALYAN KAMAL |
| Directorate General of Quality Assurance, Ministry of Defence, New Delhi | SHRI A. MITRA SHRI D. KARTIKEY (<i>Alternate</i>) |
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| Tata Steel Limited, Kolkata | DR JATIN MOHAPATRA DR RAVIKRISHNA CHATTI (<i>Alternate</i>) |
| BIS Directorate General | SHRI SANJIV MAINI, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (METALLURGICAL ENGINEERING) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)] |

Member Secretary

SHRI ASHISH PRABHAKAR WAKLE
SCIENTIST 'C'/DEPUTY DIRECTOR
(METALLURGICAL ENGINEERING), BIS

(Concluded from second cover)

Part 24 Determination of nitrogen in steel by inert gas fusion — Thermal conductivity method
(nitrogen 0.001 to 0.2 percent)

The composition of the committee responsible for the formulation of this standard is given in [Annex A](#).

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

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