***भारतीय मानक*  IS 12308 (Part 7) : 2024**

***Indian Standard***

**ढलवाँ लोहा और कच्चा लोहा के रासायनिक विश्लेषण की पद्धतियाँ**

**भाग 7 डाइमिथाइल ग्लाईऑक्सिम ग्रेवीमेट्रिक पद्धति द्वारा निकल का निर्धारण**

**(0.5 प्रतिशत से 36 प्रतिशत निकल के लिए)**

*( पहला पुनरीक्षण )*

 **Methods for Chemical Analysis of Cast Iron and Pig Iron**

**Part 7 Determination of Nickel by Dimethyl-glyoxime Gravimetric Method**

**(For Nickel 0.5 Percent to 36 Percent)**

*( First Revision )*

ICS 77.080.10

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भारतीय मानक ब्यूरो

BUREAU OF INDIAN STANDARDS

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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 7) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical analysis of Metals Sectional Committee had been approved of the Metallurgical Engineering Division Council.

This standard was first published in 1991. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

This standard is published in different parts covering methods for chemical analysis of cast iron and pig iron are as listed below. The standard (Part 7) covers determination of nickel by dimethyl-glyoxime (gravimetric) method (for nickel 0.5 to 36 percent).

The other parts in the series are:

|  |  |
| --- | --- |
| Part 1 | Determination of total carbon by thermal conductivity method |
| Part 2 | Determination of sulphur by iodimetric titration method |
| Part 3 | Determination of manganese by periodate spectrophotometric method |
| Part 4 | Determination of total carbon, graphitic carbon and combined carbon by gravimetric method |
| Part 5 | Determination of phosphorus by alkalimetric method (for phosphorus 0.01 to 0.50 percent) |
| Part 6 | Determination of silicon ( for silicon 0.1 to 6.0 percent ) |
| Part 8 | Determination of chromium by persulphate oxidation method ( for chromium 0.1 to 28 percent ) |
| Part 9 | Determination of molybdenum by thiocyanate ( spectrophotometric ) method ( for molybdenum 0.1 to 1.0 percent ) |
| Part 10 | Determination of manganese ( up to 7.0 percent ) by arsenite ( volumetric ) method |
| Part 11 | Determination of total carbon by the direct combustion volumetric method ( for carbon 1.50 to 4.50 percent) |
| Part 12 | Determination of copper by atomic absorption spectrometric method (for copper 0.01 to 0.5 percent) |
| Part 13 | Determination of magnesium by atomic absorption spectrometric method (for magnesium upto 0.1 percent) |
| Part 14 | Determination of titanium by hydrogen peroxide (spectrophotometric) method (for titanium up to 0.25 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*)’.

*Indian Standard*

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 7 DETERMINATION OF NICKEL BY DIMETHYL-GLYOXIME (GRAVIMETRIC) METHOD

(FOR NICKEL 0.5 PERCENT TO 36 PERCENT)

*( First Revision )*

**1 SCOPE**

This standard (Part 7) covers the method for determination of nickel in the range from 0.5 percent to 36 percent in alloy cast iron and pig iron.

**2 REFERENCES**

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

|  |  |
| --- | --- |
| *IS No.* | *Title* |
| IS 264 : 2005 | Nitric acid ― Specification (*third revision*) |
| IS 265 : 2021 | Hydrochloric acid – Specification (*fifth 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 the filtrate reserved under **5.3.1** reagents and distilled water shall be employed in the test.

**5 DETERMINATION OF NICKEL BY DIMETHYL-GLYOXIME (GRAVIMETRIC)**

**5.1 Outline of the Method**

After separating silicon, nickel is precipitated as nickel dimethyl-glyoximate and weighed.

**5.2 Reagents**

**5.2.1** *Concentrated Nitric Acid*, rd = 1.42 (conforming to IS 264).

**5.2.2** *Dilute Hydrochloric Acid*, 1:1 (*v/v*)

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

**5.2.4** *Hydrofluoric Acid*, 40 percent.

**5.2.5** *Potassium Bisulphate*, solid.

**5.2.6** *Potassium Pyrosulphate*, solid.

**5.2.7** *Citric Acid Solution*, 50 percent (*m/v*).

**5.2.8** *Concentrated Ammonia Solution*, rd = 0.90

**5.2.9** *Dimethyl-glyoxime Solution* (1 percent).

Dissolve 1 g of dimethy-glyoxime in 80 ml of portion 10 ml for each 10 mg of nickel expected. Neutralise the solution with ammonia solution and rectified spirit and before use make up to 100 ml with the spirit. Filter before use.

**5.3 Procedure**

**5.3.1** Transfer an appropriate quantity of the sample that will contain 20 mg to 60 mg of nickel, to a 400 ml beaker. Add 30 ml to 40 ml of dilute hydrochloric acid (1:1). Heat gently and when the reaction has subsided, add 5 ml concentrated nitric acid to oxide to the solution. Evaporate the solution to dryness and bake for 30 minutes at 120 °C. Add to it 25 ml to 30 ml of dilute hydrochloric acid (1:1) and heat for dissolution. Filter through a medium textured filter paper and wash with hot water. Preserve the filtrate.

**5.3.2** Ignite the residue in a platinum crucible at 800 °C. Cool, moisten with a few drops of dilute sulphuric acid (1:1) and add 5 ml to 10 ml hydrofluoric acid. Evaporate to dryness and fuse the residue with potassium bisulphate potassium pyrosulphate and take the mass in dilute hydrochloric acid (1:1) and mix the solution with the filtrate reserved under **5.3.1**

**5.3.3** Dilute the solution to about 400 ml with water and add 50 ml of citric acid solution neutralise with ammonia solution and acidify with dilute hydrochloric acid (1:1). Heat the solution to 90 °C and add excess of dimethyl-glyoxime solution (10 ml for every 10 mg of nickel). Neutralise the solution with ammonia solution and add 2 ml to 3 ml excess and stir well. Keep the solution at about 60 °C for one hour and the cool to room temperature, filter and wash 8 times to 10 times with cold water containing few drops of ammonia solution.

NOTE – As copper, cobalt and manganese also consume dimethy-glyoxime, additional excess of another 25 ml to 30 ml of dimethyl-glyoxime solution must be added, if any are present.

**5.3.4** Dissolve the precipitate by adding hot dilute hydrochloric acid (1:1) on the filter paper and collect the solution in the same beaker in which precipitation was carried out. Cool and dilute to 300 ml. Add 10 ml of citric acid solution, neutralize the solution with dilute hydrochloric acid (1:1). Heat the solution to 90 ºC and add dimethyl-glyoxime solution in excess, in the proportion 10 ml for each 10 ml of nickel expected. Neutralise the solution with ammonia solution and add 2 ml excess. Keep the solution at above 60 °C for one hour. Cool the solution and filter through a sintered glass crucible (No. 3) which previously dried at 120 °C and weighed to constant mass (*M1*). Wash the precipitate 10 times to 15 times with water containing few drops of ammonia solution. Dry the precipitate at 150 °C to constant mass. Cool in a desiccator and weigh (*M2*).

**5.4 Calculation**

 Nickel, percent by mass = $\frac{A}{B}$ × 20.32

where

*A* = (*M2-M1*) = mass, in g, of nickel dimethyl-glyoximate, and

*B* = mass, in g, of sample taken.

**5.5 Reproducibility**

± 0.025 at 0.5 percent nickel,

± 0.03 at 1 percent nickel,

± 0.07 at 5 percent nickel,

± 0.12 at 10 percent nickel,

± 0.17 at 15 percent nickel,

± 0.22 at 20 percent nickel,

± 0.28 at 25 percent nickel,

± 0.33 at 30 percent nickel.

**ANNEX A**

(*Foreword*)

**COMMITTEE COMPOSITION**

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

| *Organization* |  | *Representative(s)* |
| --- | --- | --- |
| CSIR - National Metallurgical Laboratory, Jamshedpur |  | Dr Sanchita Chakravarty **(*Chairperson*)** |
| Arcelor Mittal Nippon Steel, Mumbai |  | Shri Manoj Gupta |
|  Shri Kirit Tailor (*Alternate*) |
| Bhabha Atomic Research Centre, Mumbai |  | Ms Sanjukta A. Kumar |
|  | Shri M. V. Rana (*Alternate*) |
| CSIR - National Metallurgical Laboratory, Jamshedpur |  | Dr Ashok K. Mohanty (*Alternate*) |
| Defence Metallurgical Research Laboratory, Ministry of Defence, Hyderabad |  | Shri S. S. Kalyan Kamal |
| Directorate General of Quality Assurance, Ministry of Defence, New Delhi |  | Shri Kesavamoorthy M |
|  | Shri E Suman. Kumar (*Alternate*) |
| Geological Survey of India, New Delhi |  | Shri Nitin Purushottam |
|  |  Smt. Sanjukta Dey Pal (*Alternate*) |
| Hindalco Industries Limited, Mumbai |  | Shri Krishanu Mahapatra |
|  | Shri Ashutosh Acharya (*Alternate*) |
| Indian Metals and Ferro Alloys Limited, Bhubaneswar |  | Shri Dinesh Kumar Mohanty |
| JSW Steel Limited, Mumbai |  | Shri Kotrabasavaraju |
|  | Shri Marulasiddesha U. M. (*Alternate*) |
| Jawaharlal Nehru Aluminium Research Development and Design Centre, Nagpur |  |
|  | Dr Upendra Singh  |
| National Aluminium Company Limited, Bhubaneswar |  | Smt Sukla Nandi |
|  | Shri Debananda Bhattacharyya (*Alternate*) |
| National Mineral Development Corporation, Hyderabad |  | Dr Saroj Kumar Sahu |
|  |  Shri Ashish Shrivastava (*Alternate*) |
| National Test House, Kolkata |  | Dr Rajeev Kumar Upadhyay |
|  | Shri Akbar H. (*Alternate*) |
| Shriram Institute for Industrial Research, Delhi |  | Shri Dr Laxmi Rawat |
|  | Shri Puneet Kapoor (*Alternate*) |
| Research Designs and Standards Organization (RDSO), Lucknow |  | Shri Sandeep |
|  |  Smt Sunia (*Alternate*) |
| Steel Authority of India Limited - Salem Steel Plant, Salem |  | Shri L. Sivakumar |
|  | Shri Vivekanandhan G. (*Alternate*) |
| TRL Krosaki Refractories Limited, Belpahar |  | Shri S. K. Subudhi |
| Tata Steel Limited, Kolkata |  | Shri Dr Jatin Mohapatra |
|  |  Dr Ravikrishna Chatti (*Alternate*) |
| BIS Directorate General |  | Shri Sanjiv Maini, Scientist ‘F’/Senior Director and Head (Metallurgical Engineering) [Representing Director General (*Ex-officio*)] |

*Member Secretary*

Shri Ashish Prabhakar Wakle

Scientist ‘D’/Joint Director

(Metallurgical Engineering), BIS