भारतीय मानक प्रारूप

ढलवां लोहे एवं कच्चे लोहे के रासायनिक विश्लेषण की पद्धतियाँ

भाग 7 डाइमिथाइल-ग्लाइअक्सिम (भारात्मक) पद्धति से निकिल ज्ञात करना

(0.5 से 36 प्रतिशत निकिल के लिए)

(पहला पुनरीक्षण)

Draft Indian Standard

METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 7 DETERMINATION OF NICKEL BY DIMETHYL-GLYOXIME (GRAVIMETRIC) METHOD (FOR NICKEL 0.5 TO 36 PERCENT)

(First Revision)

ICS 77.080.10

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

XXXX **2022** **Price Group**

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This draft Indian Standard (Part 7) (First Revision) subject to its finalization, is to be adopted by the Bureau of Indian Standards on recommendation of the Methods of Chemical analysis of Metals Sectional Committee and approval of the Metallurgical Engineering Division Council.

Chemical analysis of cast iron and pig iron was covered in IS 228 : 1959 'Methods of Chemical analysis of pig iron, cast iron and plain carbon and low alloy steels (*revised*)'. During its second revision it was decided that a comprehensive series should be prepared for chemical analysis of all types of steels and the other covering the chemical analysis of cast iron and pig iron. Accordingly, IS 228 on revision was published in several parts covering chemical analysis of various steels only and separate series of standard under IS 12308 is being published for chemical analysis of cast iron and pig iron.

This standard was first published in 1991 in different parts covering methods for chemical analysis of cast iron and pig iron. The standard (Part 7) covers determination of nickel by dimethylglyoxime (gravimetric) method (for nickel 0.5 to 36 percent) and prescribed on the basis of experience gained during the past and reproducibility of the method was incorporated at different percent levels of nickel.

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)

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

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.

Draft Indian Standard

METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 7 DETERMINATION OF NICKEL BY DIMETHYL-GLYOXIME (GRAVIMETRIC) METHOD (FOR NICKEL 0.5 TO 36 PERCENT)

(First Revision)

1 SCOPE

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

2 REFERENCE

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

IS No.	*Title*
264 : 2005	Nitric acid — Specification (*third revision*)
265 : 2021	Hydrochloric acid – Specification (*fifth revision*)

3 SAMPLING

The sample shall be drawn and prepared as prescribed in the relevant 1ndian Standard.

4 QUALITY OF REAGENTS

4.1 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 to 60 mg of nickel, to a 400 mi beaker. Add 30-40 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 25-30 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 5-10 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 2-3 ml excess and stir well. Keep the solution at about 60°C for one hour and the cool to room temperature, filter and wash 8-10 times with cold water containing few drops of ammonia solution.

NOTE – As copper, cobalt and manganese also consume dimethy-glyoxime, additional excess of another 25-30 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 90C 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 (M_1). Wash the precipitate 10-15 times with water containing few drops of ammonia solution. Dry the precipitate at 150°C to constant mass. Cool in a desiccator and weigh (M_2).

5.4 Calculation

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

where

$A = (M_2\text{-}M_1)$ = 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.