BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

Draft Indian Standard

METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 13 DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTROMETRIC METHOD (FOR MAGNESIUM UPTO 0.1 PERCENT)

[*First Revision* of IS 12308 (Part 13)]

ICS 77.080.10

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34	Last date of comments **16 March 2023**

FOREWORD

This draft Indian Standard (Part 13) (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 hen 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 a separate series of standards under IS 12308 is being published for chemical analysis of cast iron and pig iron.

This standard was first published in 1992 in different parts covering methods for chemical analysis of cast iron and pig iron. This standard (Part 13) covers determination of magnesium by atomic absorption spectrometric method (for magnesium upto 0.1 percent). The atomic absorption spectrometric method had been prescribed in this part on the basis of inter-laboratory tests carried on the standard samples, by the various laboratories.

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 7	Determination of nickel by dimethylglyoxime (Gravimetric) method (for nickel 0.5 to 36 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 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*)’.

Draft Indian Standard

METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 13 DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTROMETRIC METHOD (FOR MAGNESIUM UPTO 0.1 PERCENT)

(First Revision)

1 SCOPE

This standard (Part 13) describes the method for determination of magnesium in pig iron and cast iron up to 0.1 percent by atomic absorption spectrometric method.

2 REFERENCES

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*
IS 264 : 2005	Nitric acid — Specification (*third revision*)
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)

3 SAMPLING

Samples shall be drawn and prepared as per the relevant Indian Standard.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OP MAGNESIUM

5.1 Outline of the Method

Sample is decomposed by treatment with hydrochloric acid and a little nitric acid. Silica is removed by dehydration and subsequent hydrofluoric acid treatment. The solution con-taming a releasing agent is aspirated in air-acetylene flame. Atomic absorption spectrometric measurements are made at 285.2 nm.

5.2 Reagents

5.2.1 *Hydrochloric Acid,* rd. = 1.16 (conforming to IS 265).

5.2.2 *Dilute Hydrochloric Acid,* 1:1 and 1:9 (*v/v*).

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

5.2.4 *Hydrofluoric Acid*, 40 percent (*m/v*).

5.2.5 Dilute Sulphuric Acid, la (*v/v*).

5.2.6 *Lanthanum Chloride Solution* (1 percent).

Dissolve 10 g of $LaCl_3$. H_2O in 100 ml hot dilute hydrochloric acid (1:1), cool and dilute to one litre.

5.2.7 *Pure Iron*

5.2.8 *Iron Background Solution*

Dissolve 1.25 g pure iron in 70 ml of hydro-chloric acid and oxidize the solution with nitric acid adding in small quantity. Add 2.5 g of sodium carbonate and dilute the solution to 250 ml.

5.2.9 *Magnesium Standard Solution* (1 ml = 1 000 µg Mg).

Dissolve 0.5 g of oxide free pure magnesium in 30 ml dilute hydrochloric acid (1:1). Transfer the solution to a 500 ml volumetric flask and dilute to mark with water and mix. Preserve the solution in a polyethylene container.

5.2.9.1 *Magnesium standard solution* (1 ml = 100 µg Mg).

Transfer 25 ml of magnesium solution (**5.2.9**) to a 250 ml volumetric flask. Dilute to the mark with water and mix.

5.2.9.2 *Magnesium standard solution* (1 ml 10 µg Mg). Transfer 25 ml of magnesium solution (**5.2.9.1**) to a 250 ml volumetric flack. Mute to the mark with water and mix.

5.2.9.3 *Magnesium standard solution* (1 ml = 1 µg Mg).

Transfer 10 ml of the magnesium standard solution (**5.2.9.2**) to a 100 ml volumetric flask. Dilute to the mark with water and mix.

5.3 Apparatus

5.3.1 *Atomic Absorption Spectrometer*

Equipped with a monochromatic radiation source such as magnesium hollow cathode lamp, a monochromator to isolate the 285.2 nm resonance line, an atomization source such as a burner and a readout device.

5.3.2 *Operating Parameters*

5.3.2.1 *Magnesium hollow cathode lamp*

5.3.2.2 *Wavelength*, 285.2 nm.

5.3.2.3 *Flame*, Air-acetylene, oxidizing; lean.

5.3.2.4 *Band pass*, 0.2/0.4 run or as specified by the manufacturer.

NOTE – Other operating parameters to be followed according to manufacturer's instructions.

5.4 Procedure

5.4.1 *Test Portion*

Weigh to the nearest 0.001 g, 0.5 g of the sample. Transfer it to a 250 ml beaker.

5.4.2 *Dissolution of the Test Portion*

5.4.2.1 Add 25 nil of dilute hydrochloric acid (1:1) when the reaction has subsided, add nitric acid in small quantities to oxidize the solution. Evaporate the solution to dryness and keep at 110°C for 30 minutes. Cool, add 30 ml of dilute hydrochloric acid (1:1) and heat to dis-solve the salts. Filter the solution through a medium textured filter paper washing 4-5 times with hot dilute hydrochloric acid (1:9) and then thoroughly with hot water. Preserve the filtrate and evaporate to a volume about 50-60 ml.

5.4.2.2 Transfer the filter with residue in platinum crucible. Incinerate the paper and ignite at 800°C. Cool and moisten the residue with water. Add 2-3 drops of dilute sulphuric acid (1:1) and 10 ml of hydrofluoric acid. Evaporate the acid till fumes of sulphur trioxide cease to evolve and ignite at 800°C for 5 minutes. Add 1 g of sodium carbonate and fuse at 1 000°C. Take the fused mass with the solution preserved in **5.4.2.1**. Filter if necessary and dilute to 100 ml in a volumetric flask.

5.4.2.3 Pipette 20 ml aliquot (**5.4.2.2**) into a 100 ml volumetric flask, add 10 ml of Lanthanum chloride solution, dilute with water to the mark and mix.

5.4.3 *Preparation of Calibration and Blank Solution*

5.4.3.1 Take six number 100 ml volumetric flask, and to each flask add 20 ml of the back-ground solution (**5.2.8**). To each of the flasks add 0, 1, 2, 5, 8 and 10 ml of standard magnesium solution (**5.2.9.3**) and 10 ml of Lanthanum chloride solution. Dilute to the mark with water and mix well.

5.4.3.2 The zero member of the above series will serve as the blank for the calibration and test solution as well.

5.4.4 *Adjustment of the Atomic Absorption Spectrometer*

Follow the instructions of the manufacturer in preparing the instrument. Switch on the instrument and the magnesium hollow cathode lamp. Fit the correct burner for air-acetylene flame and light the flame (fuel-lean). Wait for about 20 minutes for stabilization. Set the wave length at 285.2 nm. Optimize instrument response by adjusting the wavelength, fuel, air, burner and nebulizer while aspirating the highest calibration solution. As the sensitivity varies from instrument to instrument the concentration of the standard series and of the test solution should be adjusted accordingly. At the same time check the linearity of the calibration curve.

Aspirate water and one of the calibration solution repeatedly to ensure that there is no drift of absorbance. Finally aspirate water and set the absorbance to zero reading.

5.4.5 *Atomic Absorption Measurement*

5.4.5.1 Aspirate first the blank solution and then the calibration solution in increasing order. Aspirating water between each aspiration of the solution and record the absorbance reading. Then asphalt the test sample and note the absorbance. Each aspiration should be made at least three times and the average value taken. Solids which build up on the burner slit must be removed. Otherwise they will lead to decrease in sensitivity.

5.4.5.2 Prepare a calibration curve by plotting the absorbance (corrected for blank) against the concentration (µg/ml of Mg) of the calibration solutions.

5.4.5.3 Read the concentration of the test solution referring to the calibration curve pre-pared in **5.4.5.2**.

5.4.6 Calculation

$$\text{Magnesium, percent by mass} = \frac{A - B}{C \times 10}$$

where

A = concentration, in µg/ml of magnesium test solution,

B = concentration, in µg/ml of magnesium in the blank, and

C = mass, in g, of the test portion taken.