

भारतीय मानक प्रारूप
ढलवां लोहे एवं कच्चे लोहे के रासायनिक विश्लेषण की पद्धतियाँ
भाग 12 परमाण्वीय अवशोषण स्पेक्ट्रोमेट्री पद्धति द्वारा ताम्बा ज्ञात करना
(0.01 से 0.5 प्रतिशत ताम्बा के लिए)
(पहला पुनरीक्षण)

Draft Indian Standard

**METHODS OF CHEMICAL ANALYSIS
OF CAST IRON AND PIG IRON**

**PART 12 DETERMINATION OF COPPER BY ATOMIC ABSORPTION
SPECTROMETRIC METHOD (FOR COPPER 0.01 TO 0.5 PERCENT)**
(*First Revision*)

ICS 77.080.10

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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This draft Indian Standard (Part 12) (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 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 12) covers determination of copper by atomic absorption spectrometric method (for copper 0.01 to 0.5 percent). In this part, the gravimetric method for determination of copper had been replaced by atomic absorption spectrometric method.

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 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 12 DETERMINATION OF COPPER BY ATOMIC ABSORPTION
SPECTROMETRIC METHOD (FOR COPPER 0.01 TO 0.5 PERCENT)***(First Revision)***1 SCOPE**

This standard (Part 12) describes the method for determination of copper in pig iron and cast iron in the range from 0.01 to 0.5 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:

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
1070 : 1992	Reagent grade water – Specification (<i>third revision</i>)

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.

5 DETERMINATION OF COPPER**5.1 Outline of the Method**

After dissolution of the sample in a mixture of hydrochloric and nitric acid, the solution is aspirated into air-acetylene flame. Atomic absorption spectrometric measurements are made at 324.7 nm.

5.2 Reagents

5.2.1 *Dilute Nitric Acid, (1:4) (v/v).*

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

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

5.2.4 *Standard Copper Solutions*

5.2.4.1 Stock copper solution (1 ml = 1 mg Cu) Weigh to the nearest 0.000 1 g, 100 g of high purity copper (99.95 percent, minimum).

Transfer to a 400 ml beaker and dissolve in 25 ml of dilute nitric acid (1:4). Cover, after the vigorous reaction has subsided and evaporate on a water bath till crystallization starts. Dissolve the residue in water and cool. Transfer to a 1 000 ml volumetric flask, dilute to the mark and mix.

5.2.4.2 Standard copper solution (1 ml = 100 µg Cu)

Transfer 10 ml of the stock solution **5.2.4.1** into 100 ml volumetric flask, dilute to the mark and mix.

5.2.4.3 Standard copper solution (1 ml = 5 µg Cu)

Transfer 10 ml of the standard solution **5.2.4.2** into a 200 ml volumetric flask, dilute to the mark and mix. Prepare this solution, as and when required.

5.2.5 Iron of High Purity

5.3 Apparatus

5.3.1 Atomic Absorption Spectrometer

Equipped with a monochromatic radiation source such as copper hollow cathode lamp, a monochromator to isolate the 324.7 nm resonance line, an atomization source such as a burner and a read out device.

5.3.2 Operating Parameters

5.3.2.1 Copper hollow cathode lamp

5.3.2.2 Wavelength, 324.7 nm.

5.3.2.3 Flame, air-acetylene (slightly lean).

5.3.2.4 Band pass, as specified by the manufacturer.

5.4 Procedure

5.4.1 Test Portion

Weigh to the nearest 0.001 g, 1.0 g of the sample. Transfer to a 250 ml beaker.

5.4.2 Dissolution of the Test Portion Add 10 ml of dilute hydrochloric acid (1:1), followed by dropwise addition of 5 ml of nitric acid. Cover the beaker and heat gently until solvent action ceases. Evaporate to dryness. Add 5 ml of dilute hydrochloric acid (1:1) and 25 ml of water and heat gently to dissolve the salts. Cool, and transfer quantitatively to a 100 ml volumetric flask. Dilute to mark and mix.

Filter by decantation through dry inter paper, to remove graphite, silica or tungstic acid and collect the filtrate in a dry beaker, after discarding the first 5 to 10 ml of the filtrate. Use the filtrate for AAS measurement.

5.4.3 Preparation of Calibration and Blank Solutions

5.4.3.1 Weigh 10 g of iron in a 400 ml beaker. Add 100 ml of dilute hydrochloric acid (1:1). Heat gently to dissolve. Add 10 ml of nitric acid. Evaporate to dryness. Add 50 ml dilute hydrochloric acid (1:1), 250 ml of water and heat to dissolve the salts. Transfer to 500 ml volumetric flask, dilute to the mark and mix.

Transfer 50 ml aliquot of the iron solution, to each of the six 100 ml volumetric flasks. Add 0, 2.5, 5, 10, 20, 25 ml of copper standard solution **5.2.1.3** to each flask, dilute to the mark and mix.

5.4.3.2 Prepare a blank solution using same quantities of all the reagents and following the procedure as specified in **5.4.2** but without the sample.

5.4.4 Adjustment of Atomic Absorption Spectrometer

Follow the instructions of the manufacturer in preparing the instrument. Switch on the instrument and copper hollow cathode lamp. Fit the correct burner for air-acetylene flame and light the flame. Wait for about 20 minutes for stabilization. Set the wavelength at 324.7 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 the linearity of the calibration curve should be checked.

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 value. Then aspirate the test solutions and record the absorbance readings. Each aspiration should be made at least three times and the average value to be taken. Solids which build up on the burner slit must be removed, otherwise they will lead to a decrease in sensitivity.

5.4.5.2 Prepare a calibration curve by plotting the absorbance (corrected for blank) against the concentration ($\mu\text{g Cu/ml}$) of the calibration solutions.

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

5.4.6 Calculation

$$\text{Copper, percent by mass where} = \frac{A - B}{C \times 10^2}$$

where

A = concentration, in $\mu\text{g/ml}$, of copper test solution;

B = concentration, in $\mu\text{g/ml}$, of copper in the blank; and

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