

भारतीय मानक प्रारूप
ढलवां लोहे एवं कच्चे लोहे के रासायनिक विश्लेषण की पद्धतियाँ
भाग 8 परसल्फेट ऑक्सीकरण विधि द्वारा क्रोमियम का निर्धारण
(0.1 से 28 प्रतिशत क्रोमियम के लिए)
(पहला पुनरीक्षण)

Draft Indian Standard

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

**PART 8 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION
METHOD (FOR CHROMIUM 0.1 TO 28 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 8) (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 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 Indian Standards 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. This standard (Part 8) covers determination of chromium by persulphate oxidation method (for chromium 0.1 to 28 percent). In this part the persulphate oxidation method for determination of chromium in pig iron and cast iron had been updated and reproducibility of the method incorporated at different percent levels of chromium on the basis of experience gained.

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 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.

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

**PART 8 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION
METHOD (FOR CHROMIUM 0.1 TO 28 PERCENT)
(*First Revision*)**

1 SCOPE

This standard (Part 8) covers the method for determination of chromium in the range from 0.1 to 28 percent in alloy cast iron and pig iron.

2 REFERENCE

The Indian Standards listed 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 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

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

4 QUALITY OF REAGENTS

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

5 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION METHOD**5.1 Outline of the Method**

Sample is dissolved in sulphuric acid-phosphoric acid mixture and chromium (III) is oxidised to Chromium (VI) by ammonium persulphate in presence of silver nitrate as catalyst. Chromium is then reduced to Chromium (III) by excess of ferrous ammonium sulphate solution and the excess ferrous ammonium sulphate is titrated with standard potassium permanganate solution.

5.2 Reagents**5.2.1 Sulphuric Acid-Phosphoric Acid Mixture**

Add slowly, while stirring, 160 ml of sulphuric acid to 600 ml water. Cool and add 80 ml of phosphoric acid.

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

5.2.3 Dilute Hydrofluoric Acid, 40 percent (v/v).

5.2.4 Dilute Sulphuric Acid, 1:5 (v/v).

5.2.5 Potassium Bisulphate/Potassium Pyrosulphates, Solid

5.2.6 Silver Nitrate, 0.5 percent (m/v).

5.2.7 Ammonium Persulphate, 15 percent (m/v).

5.2.8 Potassium Permanganate Solution, 0.1 N.

Dissolve 3.2 g of potassium permanganate in one litre of water and keep in the dark for several days. Filter the solution through a sintered glass crucible and store in a well stoppered dark coloured bottle. Standardise the solution as follows:

Dissolve 0.134 g of extra pure sodium oxalate crystals, dried for 1 hour at 105°C in a 200 ml of dilute sulphuric acid (1:50). Heat to 70°C. Titrate with potassium permanganate solution until one drop produces a permanent pink colouration.

$$[1 \text{ ml of } 0.1 \text{ N KMnO}_4 = 0.0067 \text{ g of Na}_2(\text{COO})_2]$$

5.2.9 Dilute Hydrochloric Acid, 1:3 (v/v).

5.2.10 Standard Ferrous Ammonium Sulphate, (0.1 N).

Dissolve 40 g of ferrous ammonium sulphate in 500 ml of cold dilute sulphuric acid (5 percent) and dilute to one litre with the same (*see* Note 1). Standardize the solution as follows:

Transfer 180 ml of water, 12 ml of dilute sulphuric acid (1:1) and 5 ml of phosphoric acid into a 500ml Erlenmeyer flask Add 20 ml of ferrous ammonium sulphate solution and finally add 1 to 2 drops of 1,10 phenanthroline indicator solution. Titrate the ferrous ions with 0.1 N KMnO₄ standard solution (**5.2.8**) while swirling the flask. Continue until the pink colour changes to clear green and persists for at least 60 s.

Calculate the normality of ferrous ammonium sulphate solution as follows:

$$\text{Normality} = \frac{AB}{C}$$

where

A = normality of potassium permanganate solution (*see* **5.2.8**);

B = volume, in ml, of potassium permanganate solution used; and

C = volume, in ml, of ferrous ammonium sulphate solution.

NOTE – The solution, if saturated with carbon dioxide will be more stable.

5.3 Procedure

5.3.1 Take the quantity of sample depending upon the chromium content as follows:

2 g for chromium content between 0.1 and 2.0 percent,

1 g for chromium content between 2.0 and 10.0 percent,

0.5 g for chromium content greater than 10.0 percent

5.3.2 Take the amount of sample as prescribed in **5.3.1** in a 400-ml beaker. Add 60 ml of the sulphuric acid-phosphoric acid mixture and heat gently to dissolve the sample. When the reaction has subsided, add concentrated nitric acid dropwise to oxidise the solution.

NOTE – For samples rich in silicon, add few drops of hydrofluoric acid and evaporate the solution to light fuming.

5.3.3 Cool the solution and dilute to 100 ml with water. Filter through a medium textured filter paper washing thoroughly with hot water. Reserve the filtrate.

5.3.4 Ignite the residue in a platinum crucible at about 900°C. Cool, add few drops of dilute sulphuric acid and 5 to 10 ml hydrofluoric acid. Evaporate to dryness and fuse the residue with potassium bisulphate/potassium pyrosulphate and extract the fused mass with dilute sulphuric acid (1:5). Add to the filtrate preserved in **5.3.3**.

5.3.5 Transfer the solution (**5.3.4**) to a 500-ml conical flask, dilute to about 300 ml and heat to boiling.

Add 15 ml of silver nitrate solution and 30 ml of ammonium persulphate solution till permanganate colour is formed. When the manganese content is very low add 1 ml of potassium permanganate solution. Boil the solution for 10 minutes and decompose the permanganic acid by adding dilute hydrochloric acid dropwise until the pink colour disappears. Boil to expel chlorine. Cool the solution to room temperature. During boiling keep the volume of the solution to 300 ml by adding water from time to time.

5.3.6 To the solution (**5.3.5**), add slight excess of ferrous ammonium sulphate solution from a burette. Titrate immediately the excess of Fe (II) with standard potassium permanganate solution. At the 'end point' the pink colour should persist for one minute. Note the volume of the titrant (*A*). Now boil the solution to destroy the pink colour. Cool and add the same volume of ferrous ammonium sulphate solution as added before and titrate the Fe (II) to the same pink colour as in the first titration. Note the volume of the titrant (*B*).

5.3.7 Calculation

$$\text{Chromium, Percent by mass} = \frac{(B - A) \times 0.01734 \times 100 \times N}{C}$$

where,

B = volume, in ml, of KMnO₄ equivalent to the total volume of ferrous sulphate added;

A = volume, in ml, of KMnO₄ equivalent to the excess ferrous ammonium sulphate;

N = normality of KMnO₄ solution; and

C = mass, in g, of the sample taken.

5.4 Reproducibility

± 0.03 at 0.25 percent chromium

± 0.04 at 0.50 percent chromium

± 0.06 at 1.0 percent chromium

± 0.10 at 2.5 percent chromium

± 0.16 at 5 percent chromium

± 0.23 at 10 percent chromium

± 0.30 at 15 percent chromium

± 0.34 at 20 percent chromium

± 0.40 at 25 percent chromium

± 0.47 at 28 percent chromium