For Comments Only

BUREAU OF INDIAN STANDARDS

***Draft* Revision**

TO

IS 228 (Part 6): METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 6 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION METHOD (FOR CHROMIUM ≥ 0.1 PERCENT)
(Fourth Revision)

Last date for receipt of comments is XXXX

भारतीय मानक प्रारूप

इस्पात के रासायनिक विश्लेषण की पद्धतियाँ

भाग 6 XXXX

(चौथा पुनरीक्षण)

Draft Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF STEELS

PART 6 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION METHOD (FOR CHROMIUM ≥ 0.1 PERCENT)

(Fourth Revision)

ICS 77.080.20

BUREAU OF INDIAN STANDARDS
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XXXX **2023** **Price Group**

Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This draft Indian Standard (Part 6) (Fourth 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.

This standard was first published in 1952 and subsequently revised in 1959, 1974 and 1987, covered the chemical analysis of plain carbon and low alloy steels, along with pig iron and cast iron. It was revised again to make it comprehensive in respect of steel analysis and to exclude pig iron and cast iron which were being covered in separate standards. During its second revision the standard has been split up in several parts. During third revision, following modifications were made:

a) scope of the method was modified by lowering the limit for determination of chromium from 0.5 to 0.1 percent;
b) only one method was prescribed for the correction in the titration of chromium for dilution effect and colour interference; and
c) inclusion of reproducibility of the method at the ,various levels of chromium content.

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

This part covers method for the determination of chromium by persulphate oxidation method. The other parts of this series are:

Part 1	Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent)
Part 2	Determination of manganese in plain carbon and low alloy steels by arsenite method
Part 3	Determination of phosphorus by alkalimetric method
Part 4	Determination of total carbon by gravimetric method (for carbon greater than or equal to 0.1 percent)
Part 5	Determination of nickel by dimethyl glyoxime (gravimetric) method (for nickel greater than or equal to 0.1 percent)
Part 7	Determination of molybdenum by alpha benzoinoxime method (for molybdenum 1 percent and not containing tungsten)
Part 8	Determination of silicon by gravimetric method (for silicon 0.05 to 5.00 percent)

Part 9 Determination of sulphur in plain carbon steels by evolution method (for sulphur 0.01 to 0.25 percent)

Part 10 Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum 0.01 to 1.5 percent)

Part 11 Determination of silicon by reduced molybdosilicate spectrophotometric method in carbon steels and low alloy steels (for silicon 0.01 to 0.05 percent)

Part 12 Determination of manganese by periodate spectrophotometric method in plain carbon, low alloy and high alloy steels (for manganese 0.01 to 5.0 percent)

Part 13 Determination of arsenic

Part 14 Determination of carbon by thermal conductivity method (for carbon 0.005 to 2.000 percent)

Part 15 Determination of copper by thiosulphate iodide method (for copper 0.05 to 5 percent)

Part 16 Determination of tungsten by spectrophotometric method (for tungsten 0.1 to 2 percent)

Part 17 Determination of nitrogen by thermal conductivity method

Part 18 Determination of oxygen by instrumental method

Part 19 Determination of nitrogen by steam distillation

Part 20 Determination of carbon and sulphur by infrared absorption method

Part 21 Determination of copper by spectrometric method (for copper 0.02 to 0.5 percent)

Part 22 Determination of total hydrogen in steel by thermal conductivity method (hydrogen 0.1 ppm to 50 ppm)

Part 23 Determination of total nitrogen in steel by optical emission spectrometer (nitrogen 0.002 to 1.0 percent)

Part 24 Determination of nitrogen in steel by inert gas fusion – Thermal conductivity method (nitrogen 0.001 to 0.2 percent)

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 FOR CHEMICAL ANALYSIS OF STEELS

PART 6 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION METHOD (FOR CHROMIUM ≥ 0.1 PERCENT)

(Fourth Revision)

1 SCOPE

This standard (Part 6) covers the persulphate oxidation method for determination of chromium content of low alloy and high alloy steels containing more than or equal to 0.1 percent chromium. This method is not applicable for steels containing tungsten.

2 REFERENCES

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

IS No	*Title*
IS 264 : 2005	Nitric acid – Specification (*third revision*)
IS 1070 : 1992	Reagent grade water – Specification (*third revision*)

3 SAMPLING

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

4 QUALITY OF REAGENTS

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

5 DETERMINATION OF CHROMIUM BY PERSULPHATE OXIDATION METHOD

5.1 Outline of the method

After dissolution of the sample in dilute sulphuric acid and phosphoric acid mixture and further treated with nitric acid, chromium, manganese and (vanadium if present) are oxidized by ammonium persulphate in presence of silver nitrate as catalyst. Permanganic acid is then destroyed by dilute hydrochloric acid. Chromium is reduced by ferrous ammonium sulphate and excess of ferrous ammonium sulphate is back titrated with standard potassium permanganate solution.

5.2 Reagents

5.2.1 *Phosphoric Acid - Sulphuric Acid Mixture*

To 600 ml of water, add continuously 165 ml of concentrated sulphuric acid (rd = 1.84) and 132 ml of phosphoric acid (rd = 1.75). Mix, cool and dilute to 1 litre.

5.2.2 *Concentrated Nitric Acid*

Relative density = 1.42 (conforming to IS 264).

5.2.3 *Silver Nitrate Solution* – 0.5 percent (*m/v*).

Dissolve 5 g of silver nitrate crystals in water and dilute to 1 litre.

5.2.4 *Ammonium Persulphate Solution*

Dissolve 15 g of ammonium persulphate in 100 ml of water. Use a freshly prepared solution.

5.2.5 *Potassium Permanganate Solution* – 1 percent (*m/v*).

5.2.6 *Dilute Hydrochloric Acid* – 1:3 (*v/v*).

Dilute 250 ml of concentrated hydrochloric acid (rd = 1.16) to 1 litre.

5.2.7 *Standard Ferrous Ammonium Sulphate Solution* – Approximately 0.1 N.

Dissolve 40 g of ferrous ammonium sulphate in sulphuric acid (5 percent) and dilute to 1 litre. Filter, if necessary, and keep in a stoppered glass bottle. Standardize against standard potassium permanganate solution (given under **5.2.8**) every time it is used.

5.2.8 *Standard Potassium Permanganate Solution* – Approximately 0.1 N.

Dissolve 3.2 g of potassium permanganate crystals in 1 000 ml of water, stir and allow to stand in a closed vessel for 24 hours. Filter, through a sintered glass crucible and keep in an amber-coloured glass bottle. Standardize the solution as follows:

Dissolve 0.134 g of sodium oxalate crystals, dried for 1 hour at 105°C in 200 ml of dilute sulphuric acid (1:50). Heat to 70°C and titrate with potassium permanganate solution until one drop produces a permanent pink colouration. [1 ml of potassium permanganate solution (0.1 N) = 0.006 7 g of sodium oxalate].

5.3 Procedure

5.3.1 Take 2 g of sample (for chromium less than 2 percent) and 0.2 to 0.5 g of sample for high alloy steels in a wide mouth conical flask. Add 50 ml of phosphoric acid-sulphuric acid mixture. Heat the flask to decompose the sample. Oxidize black residue by addition of concentrated nitric acid dropwise and heating the solution simultaneously till all carbides are decomposed and brown fumes are expelled. Dilute to 300 ml with hot water.

5.3.2 Add a few pieces of glass beads, heat the solution to boiling and add 20 ml of silver nitrate solution and 20 ml of ammonium persulphate solution adding little at a time and continue boiling till the permanganate colour develops fully (volume should be maintained at 300 ml by addition

of hot water, if necessary and also boiling should be a period of 8-10 minutes). It should be ensured that sufficient persulphate is added. Wash the sides of the conical flask with water. If the colour does not develop add a few drops of potassium permanganate solution till the pink colour develops.

5.3.3 Add dilute hydrochloric acid dropwise to the boiling solution till permanganic acid colour is destroyed. Boil for 10 minutes more. Cool and add a known volume of standard ferrous ammonium sulphate solution until an excess of at least 5 ml is present. Titrate back with dropwise addition of standard potassium permanganate solution to a permanent pink end point which persists for 30-40 seconds.

5.3.4 In presence of vanadium, titrate carefully to a pink end point which persists for at least. 30 to 40 seconds, to ensure complete re-oxidation of the vanadium.

5.3.5 The titration should be corrected for dilution effect and colour interference. The correction may be made by the following method:

5.3.5.1 Add same amount of ferrous ammonium sulphate as used for the sample, to the already titrated solution. Titrate with standard potassium permanganate to pink end point which lasts for 30 to 40 seconds.

5.4 Calculation

5.4.1 Calculate the chromium content of the steel as follows:

$$\text{Chromium, percent} = \frac{(AB - C)\, D \times 0.017\ 33 \times 100}{E}$$

where

A = volume in ml of standard ferrous ammonium sulphate solution added,

B = volume in ml of standard potassium permanganate solution equivalent to 1 ml of ferrous ammonium sulphate solution,

C = volume in ml of standard potassium permanganate solution required for titration, corrected for the blank,

D = normality of standard potassium permanganate solution, and

E = mass in g of the sample taken for the test.

5.4.2 *Reproducibility*

a) ± 0.025 percent at 0.1 to 0.5 percent chromium,

b) ± 0.036 percent at 0.5 to 1 percent chromium,

c) ± 0.l20 percent at 1 to 5 percent chromium, and

d) ± 0.20 percent for chromium 5 percent and above.