

भारतीय मानक मसौदा  
रासायनिक उद्योगों के लिए क्रोमाइट — विशिष्टि

(IS 4737 का दूसरा पुनरीक्षण)

*Draft Indian Standard*  
**Chromite for Chemical Industries — Specification**  
(*Second Revision of IS 4737*)

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ICS 71.060.20

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

(*formal clauses will be added later*)

This standard was first published in 1968 and subsequently revised in 1982. In the first revision, major changes were effected in the methods of determination of silica, alumina, chromium, iron, lime & magnesium and the method for the preparation of sample.

In this second revision, the standard has been revised based on the technological advancements that have taken place since the last publication of the standard. Also, reference clause has been incorporated and Packing & Marking clause has been updated.

Chromite is the main chromium bearing mineral of commercial importance. The mineral chromite has the composition  $\text{FeO} \cdot \text{Cr}_2\text{O}_3$ , with a theoretical content of 68 percent  $\text{Cr}_2\text{O}_3$ , and 32 percent FeO by mass. It is a group of cubic minerals known as spinel's which is represented by the general formula  $\text{XO} \cdot \text{Y}_2\text{O}_3$ , where 'X' is a divalent metal ion usually, iron, calcium, magnesium or manganese and Y is the trivalent metallic ion usually, iron, chromium or aluminium. In natural chromite deposits, replacement of chromium and iron by other elements usually occurs to some extent, chromium being replaced by aluminium and trivalent iron, and iron by magnesium. Consequently, commercial chrome ore rarely contains more than 50 to 55 percent chromium and some containing up to 20 percent of both  $\text{Al}_2\text{O}_3$  and MgO.

Chromite finds extensive use in the metallurgical refractories and chemical industries, the respective use depending on the chemical composition. The chemical use of chromite is almost entirely in the primary production of sodium dichromate, and other chromium salts are subsequently produced. As vanadium is present in chromite in small amount, it is counted as chromium in this standard, and no separate method has been given for the estimation of vanadium.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off 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 the same as that of the specified value in this standard.

*Draft Indian Standard*  
**CHROMITE FOR CHEMICAL INDUSTRIES — SPECIFICATION**  
*(Second Revision)*

**1 SCOPE**

This standard prescribes the requirements and the methods of sampling and test for chromite for chemical industries.

**2 REFERENCES**

The standards given below 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 revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the standards:

<i>IS No</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification ( <i>fourth revision</i> )
IS 2109 : 1982	Methods of sampling dolomite, limestone and other allied materials ( <i>first revision</i> )

**3 REQUIREMENTS****3.1 Description**

The material shall be either in the form of lumps or concentrates free from dirt and other foreign matter.

**3.2** The material, when tested according to the methods prescribed in Annex A shall comply with the requirements specified in Table 1. Reference to the relevant clauses of Annex A is given in col (4) of the table.

**Table 1 Requirements for Chromite for Chemical Industries**

SI No.	Characteristic (On Dry Basis)	Requirement	Method of Test, Ref to CI No. in Annex A
(1)	(2)	(3)	(4)
i)	Chromic oxide ( as Cr <sub>2</sub> O <sub>3</sub> ), percent by mass, <i>Min</i>	44.0	<b>A-3</b>
ii)	Total iron (as FeO), percent by mass, <i>Max</i>	20.0	<b>A-3</b>
iii)	Alumina (as Al <sub>2</sub> O <sub>3</sub> ), percent by mass, <i>Max</i>	14.0	<b>A-4</b>
iv)	Silica (as SiO <sub>2</sub> ), percent by mass, <i>Max</i>	7.0	<b>A-5</b>
v)	Lime (as CaO), percent by mass, <i>Max</i>	3.0	<b>A-5</b>
vi)	Magnesia (as MgO), percent by mass, <i>Max</i>	14.0	<b>A-5</b>

**4 PACKING AND MARKING****4.1 Packing**

Unless otherwise agreed to between the purchaser and the supplier, the material shall be supplied in open wagons.

**4.2 Marking**

A good sized metallic or cardboard label bearing the following information with suitable paint or ink shall be conspicuously displayed on the carrier and also placed inside:

- a) Name of the material;
- b) Mass of the material;

- c) Lot number; and
- d) Manufacturer's name and recognized trade-mark, if any.

#### 4.2.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

### 5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in IS 2109. However, the number of tests and criteria for conformity shall be as prescribed in 5.2 and 5.3 respectively.

#### 5.2 Number of Tests

Tests for determining chromic oxide (as Cr<sub>2</sub>O<sub>3</sub>), shall be made on each of the individual samples separately. Tests for all other requirements shall be made on the composite sample only.

#### 5.3 Criteria for Conformity

##### 5.3.1 *For Individual Samples*

For chromic oxide tested on the individual samples, the mean and the range of test results shall be computed as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of individual test results}}{\text{Number of test results}}$$

Range (*R*) = difference between the minimum and the maximum test results.

The lot shall be declared to be in conformity with chromic oxide requirement if the value of expression ( $\bar{X} - 0.6 R$ ) is not less than the specified minimum value.

##### 5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements tested on the composite sample, the test results in respect of each characteristic shall satisfy the corresponding specified requirements.

## ANNEX A

(Clause 3.2)

## ANALYSIS OF CHROMITE FOR CHEMICAL INDUSTRIES

## A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## A-2 PREPARATION OF THE SAMPLE

## A-2.1 Procedure

Crush the representative sample so as to pass through 150 micron IS Sieve. Powder about 10 g to 15 g of the sample so prepared in an agate mortar with agate pestle till 100 percent of the sample pass through 63 micron IS Sieve. This sample should be taken for chemical analysis.

**A-2.1.1** Free moisture in the sample may be determined separately on 1 g sample drying at  $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  for 2 h.

## A-3 DETERMINATION OF CHROMIUM AND IRON

## A-3.1 Outline of the Method

The sample is fused with sodium peroxide in nickel crucible. Chromium is converted to dichromate and estimated with the addition of excess ferrous ammonium sulphate in acidified solution which is back titrated with standard potassium permanganate solution.

## A-3.2 Reagents

**A-3.2.1** *Sodium Peroxide* — Powder

**A-3.2.2** *Dilute Sulphuric Acid* — 1: 1 (v/v)

**A-3.2.3** *Ferrous Ammonium Sulphate Solution* — 0.1 N

**A-3.2.4** *Standard Potassium Permanganate Solution* — 0.1 N

**A-3.2.5** *Dilute Hydrochloric Acid* — 1: 1 (v/v)

**A-3.2.6** *Orthophosphoric acid*

## A-3.3 Procedure

Weigh accurately 0.5 g of the prepared sample in a nickel crucible of about 30 ml capacity. Add and mix thoroughly with about 4 g of sodium peroxide with a dry glass rod. Heat the crucible over a flame till the mixture inside melts. Rotate the crucible gently with a pair of tongs to mix the contents well. Then keep the crucible over the flame at cherry red heat for about 5 min. Remove and allow the crucible to cool down. Introduce the crucible into a 400 ml beaker containing about 100 ml to 150 ml of water and covered with a watch glass. The crucible should submerge under water. Slowly boil till evolution of oxygen gas ceases and peroxide is destroyed. Remove the crucible and wash down the adhering solution and decomposed mass into the same beaker, taking help of policeman. Cool the solution to room temperature.

**A-3.3.1** Filter through No. 40 Whatman filter paper or equivalent. Wash the residue with water till free from chromate. Dissolve the residue in hydrochloric acid of minimum quantity and precipitate with sodium peroxide. Boil off oxygen, filter and wash. Six to eight washings should be adequate. Combine the filtrates and transfer into 250 ml standard measuring flask and make up to volume.

**A-3.3.2** Take 50 ml of the solution, acidify with dilute sulphuric acid and add 5 ml more. Add 50 ml of ferrous ammonium sulphate solution. Add 5 ml orthophosphoric acid and titrate against standard potassium permanganate

solution to the grey end point. Carry out a blank titration 50 ml of the ferrous ammonium sulphate solution with standard potassium permanganate solution.

#### A-3.3.3 Calculation

$$\text{Chromic oxide (as Cr}_2\text{O}_3\text{), percent by mass} = \frac{25.32 \times N (V_1 - V_2)}{M}$$

where

$N$  = normality of standard potassium permanganate solution;

$V_1$  = volume in ml, of standard potassium permanganate solution required for the blank;

$V_2$  = volume in ml, of standard potassium permanganate solution required for the titration; and

$M$  = mass in g, of the prepared sample taken for the test.

**A-3.4** Treat the residue on the filter paper with hot dilute hydrochloric acid till entirely dissolved. Transfer and dilute to about 200 ml. Add 3 g to 4 g of ammonium chloride. Heat to boil and add ammonia drop wise till alkaline. Boil for 4 min to 5 min. Allow to settle. Filter through No. 41 Whatman filter paper or equivalent. Wash with hot 0.5 percent ammonium nitrate solution and finally twice with hot water.

**A-3.4.1** Dissolve residue on filter paper in hot dilute hydrochloric acid and transfer into a beaker. Cool and transfer into a 250 ml standard measuring flask. Make up to volume.

#### A-3.5 Determination of Iron

##### A-3.5.1 Outline of the Method

Ferric iron is reduced with stannous chloride excess of which is oxidized by mercuric chloride. The reduced iron is titrated against standard potassium dichromate solution using sodium diphenylamine sulphonate solution as indicator.

##### A-3.5.2 Reagents

###### A-3.5.2.1 Stannous chloride solution

Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is complete. Cool, add a few pieces of granulated tin and preserve the solution in an air-tight bottle.

**A-3.5.2.2 Mercuric chloride solution** — Prepare a saturated solution of mercuric chloride in water.

###### A-3.5.2.3 Sulphuric phosphoric acid mixture

Add slowly 150 ml of concentrated sulphuric acid to 700 ml of water with continuous stirring. Add to this solution 150 ml of syrupy phosphoric acid. Destroy any oxidizable impurities by adding potassium permanganate solution (0.1 N) drop by drop until the pink colour of permanganate persists. Cool the solution.

###### A-3.5.2.4 Sodium diphenylamine sulphonate indicator solution

Dissolve 0.2 g of the reagent in 100 ml of hot water.

###### A-3.5.2.5 Standard potassium dichromate solution — 0.1 N

Dry pure potassium dichromate at 140 °C to 150 °C for one hour. Cool in a desiccator and transfer exactly 4.904 g to a one litre volumetric flask carefully through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the salt by shaking until the solution is complete. Make up to the mark and mix well.

##### A-3.5.3 Procedure

Take 100 ml from solution **A-3.4.1** in a 500 ml conical flask. Add 5 ml concentrated hydrochloric acid. Heat to boil. Add stannous chloride solution drop wise till the solution is colourless. Add two to three drops of stannous chloride in excess. Cool the solution quickly to room temperature.

**A-3.5.4** Add 10 ml of mercuric chloride solution at a time and stir the solution. Silky white precipitate of mercurous chloride appears. Allow to stand for 5 min. Add 15 ml of sulphuric-phosphoric acid mixture. Dilute to about 200 ml to 250 ml with cold water. Add three to four drops of indicator solution. Titrate with standard dichromate solution till colour of the solution changes to stable blue violet.

**A-3.5.5 Calculation**

$$\text{Total iron (as Fe}_2\text{O}_3\text{), percent by mass} = \frac{A \times B \times 7.985}{C}$$

where

*A* = volume of standard potassium dichromate solution required;

*B* = normality of standard potassium dichromate solution; and

*C* = mass in g, of the sample taken for the test.

## **A-4 DETERMINATION OF SILICA AND ALUMINA**

### **A-4.1 General**

The procedure involves decomposition of finely powdered chromite sample with per chloric acid. The decomposed material is taken up with dilute hydrochloric acid filtered and washed. Silica is determined by hydrofluorization. Hydroxides of iron, aluminium and chromium are then precipitated out with ammonia, filtered and washed. Calcium and magnesium are determined in the filtrate. Residue is redissolved in hydrochloric acid. Iron is precipitated with addition of sodium peroxide. Aluminium is determined from the filtrate after addition of ammonium nitrate.

#### **A-4.1.1 Procedure**

Take 0.25 g of finely agated sample ( -0.63 micron IS Sieve) in a 250 ml beaker. Add 10 ml water and 25 ml of perchloric acid. Digest at low heat on a hot plate. Then most of black particles disappear, increase heat till copious fumes start (*see* Note). Extract with 20 ml dilute hydrochloric acid and digest. Filter through No. 42 Whatman filter paper transferring the entire residue and wash on filter paper with hot water till free from chloride. Collect the filtrate in a 400 ml beaker.

### **A-4.2 Determination of Silica**

#### **A-4.2.1 Outline of the Method**

The residue is ignited and silica is determined by hydrofluorization in presence of 3 drops to 4 drops of sulphuric acid.

#### **A-4.2.2 Reagents**

**A-4.2.2.1 Perchloric acid** — 70 percent (v/v)

**A-4.2.2.2 Hydrofluoric acid** — 48 percent (v/v)

**A-4.2.2.3 Dilute sulphuric acid** — 18 N

#### **A-4.2.3 Procedure**

Take the residue as in **A-4.1.1** in a previously ignited and weighed platinum crucible of about 25 ml capacity. Slowly burn off filter paper. Then ignite in a furnace up to a temperature of 1 000 °C. Remove from the furnace, cool in a freshly charged desiccator and weigh. Repeat till mass is constant.

**A-4.2.4** Add 3 drops to 4 drops of dilute sulphuric acid and 5 ml hydrofluoric acid. Heat on a hot plate till dry. Ignite slowly to 1 000 °C and weigh. Repeat hydrofluorization till mass of the crucible is constant. Take final mass.

Fuse the residue with a little sodium carbonate, take up with dilute hydrochloric acid and mix with the filtrate as in **A-4.1.1**

NOTE — In case black particles of undecomposed chromite persist, dilute with water, decant off the supernatant solution and retreat the particles with perchloric acid in the original beaker. Complete decomposition should not take more than 5 h to 6 h. Alternately, decompose 0.25 g of chromite by fusing with Na<sub>2</sub>CO<sub>3</sub> (3 g), take up with dilute hydrochloric acid, evaporate and bake. Extract with 20 ml dilute HCl and proceed as in **A-4.1.1**.

**A-4.2.5 Calculation**

$$\text{Silica (as SiO}_2\text{), percent by mass} = \frac{M_2 - M_3}{M_1} \times 100$$

where

$M_2$  = mass in g, of the residue obtained in **A-4.2.3**;

$M_3$  = mass in g, of the residue obtained after hydrofluorization in **A-4.2.4**; and

$M_1$  = mass in g, of sample taken for test in **A-4.1.1**.

**A-4.3 Determination of Alumina****A-4.3.1 Outline of the Method**

Alumina is determined by separating as sodium aluminate from the solution of mixed oxides and precipitating as aluminium hydroxide with ammonium nitrate and igniting to aluminium oxide.

**A-4.3.2 Reagents**

**A-4.3.2.1 Dilute hydrochloric acid** — 5 N

**A-4.3.2.2 Ammonium chloride** — solid

**A-4.3.2.3 Ammonium nitrate** — solid

**A-4.3.2.4 Dilute ammonium hydroxide** — 8 N

**A-4.3.2.5 Dilute sodium hydroxide solution** — 10 percent

**A-4.3.2.6 Sodium peroxide** — solid

**A-4.3.3 Procedure**

Dilute the filtrate obtained in **A-4.1.1** to about 200 ml. Heat to boil after adding 4 g of ammonium chloride. Add with stirring dilute ammonium hydroxide till a faint ammoniacal smell is obtained. Boil for a few minutes. Allow the precipitate to settle, filter through No. 41 Whatman filter paper transferring bulk of the precipitate on the filter and wash with hot water 6 or 7 times. Dissolve the precipitate in the same beaker with dilute hydrochloric acid and repeat precipitation of hydroxides. Wash with hot water collecting the filtrate in the same beaker.

**A-4.3.4** Take back the precipitate in the original beaker dissolving in hot dilute hydrochloric acid. Nearly neutralize with freshly prepared dilute sodium hydroxide solution. Add 1 g to 2 g sodium peroxide. Dilute to about 200 ml. Heat to boil till evolution of oxygen ceases. Filter through No. 41 Whatman filter paper or equivalent. Repeat precipitation. Wash precipitate with hot water.

**A-4.3.5** Treat the combined filtrate containing sodium aluminate and sodium chromate as in **A-4.3.4** with 4 g to 5 g of ammonium nitrate with continuous stirring and precipitate aluminium hydroxide. Filter through No. 41 Whatman filter paper. Repeat precipitation. Wash precipitate on filter paper with hot 0.5 percent ammonium nitrate solution till free from chloride. Ignite precipitate on  $\text{Al}_2\text{O}_3$  at about 1 000 °C.

**A-4.3.6 Calculation**

$$\text{Alumina (as Al}_2\text{O}_3\text{, ) percent by mass} = \frac{100 \times M_1}{M}$$

where

$M_1$  = mass in g, of the ignited residue, and

$M$  = mass in g, of the material taken for test.

**A-5 DETERMINATION OF LIME AND MAGNESIA****A-5.1 Outline of the Method**

Combined filtrate as obtained in **A-4.3.3** is boiled down to less than 100 ml. Aliquots are taken for determination of calcium and magnesium complexometrically.

### **A-5.2 Reagents**

**A-5.2.1 Dilute Hydrochloric Acid** — 5 N

**A-5.2.2 Potassium Hydroxide Solution** — approximately 10 N

The solution should be freshly prepared from AR quality potassium hydroxide.

**A-5.2.3 Buffer Solution (pH 10)**

Dissolve 70 g of ammonium chloride in 200 ml of water, add 570 ml of concentrated ammonia (density 0.88 to 0.90) and dilute to 1 litre.

**A-5.2.4 Patton and Keeder's Indicator**

Mix 0.1 g of 2 hydroxy 1-(2-hydroxy-4-sulpho-1 naphthalzo)-3-naphthoic acid with 10 g of potassium chloride and grind into a fine mixture in a glass mortar. Preserve in a well stoppered dry amber coloured glass bottle.

**A-5.2.5 Erio Chrome Black T Indicator**

Dissolve 0.5 g of Erio Chrome Black T indicator in 100 ml of rectified spirit and preserve in amber coloured glass bottle. This indicator should be freshly prepared preferably.

**A-5.2.6 Standard EDTA Solution**

Weigh 7.444 g of disodium ethylene diamine tetracetate dihydrate (EDTA) and dissolve in deionized water. Make up the volume to one litre. Mix thoroughly. The solution is 0.02 M.

**A-5.2.7 Potassium Cyanide and Hydroxylamine Hydrochloride Pure**

**A-5.2.8 Deionized Water**

### **A-5.3 Determination of Calcium Oxide**

**A-5.3.1 Procedure**

The combined filtrate as in **A-4.3.3** is boiled down to less than 100 ml cooled and made up to volume 100 ml in a standard measuring flask. Take two portions of 25 ml each in two conical flask or white porcelain dish of 100 ml capacity. Add about 5 ml of potassium hydroxide solution to one flask. Add about 0.2 g to 0.4 g P and R indicator and quickly titrate till the colour changed to violet blue. Note the volume of EDTA consumed. Add the same EDTA volume less 1.0 ml directly to the second flask. Add 5 ml of potassium hydroxide solution and about 50 mg each of potassium cyanide and hydroxylamine hydrochloride. Add 0.4 g indicator and immediately titrate with continuous stirring until no further colour changes and the test solution is clear blue without any tinge of violet. Volume of EDTA consumed in the final titration should be taken for calculating CaO percent (**A-5.3.2**).

**A-5.3.2 Calculation**

$$\text{Calcium oxide (as CaO), percent by mass} = \frac{0.11216 \times V_1}{M}$$

where

$V_1$  = volume in ml, of 0.02 M EDTA solution required for final titration; and

$M$  = mass in g, of the material contained in the aliquot of solution taken for the titration.

### **A-5.4 Determination of Magnesia**

**A-5.4.1 Procedure**



Take 25 ml aliquot from **A-5.2.1** in a white porcelain dish of 100 ml capacity. Add 25 ml of ammonium chloride and ammonium hydroxide buffer solution and a few drops of Erio Chrome Black T and titrate with EDTA solution till the colour changes from wine red to indicator blue.

**A-5.4.2** *Calculation*

$$\text{Magnesium (as MgO), percent by mass} = 0.08062 \times \frac{V_2 - V_1}{M}$$

where

$V_2$  = volume of 0.02 M EDTA solution required for the titration,

$V_1$  = volume of 0.02 M EDTA solution required for the sample solution, and

$M$  = mass in g, of the material contained in the aliquot of solution taken for the titration.