

कैल्शियम क्लोराइड — विशिष्टि
(तीसरा पुनरीक्षण)

Calcium Chloride — Specification
(Third Revision)

ICS 71.060.50

© BIS 2024



भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

July 2024

Price Group 9

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1958 and subsequently revised in 1967 and 1984. Calcium chloride, technical, hydrous and anhydrous were covered in IS 1334 and IS 1314 respectively. The two standards were amalgamated and revised in 1967 and consequently IS 1334 was withdrawn. Analytical reagent grades for both were also added in the first revision. In the second revision, with the addition of the fused calcium chloride, three types of the material were covered. Three grades have been specified for the hydrous material. The two grades which were prescribed for anhydrous material in the first revision were merged into one.

In this revision, instrumental test methods for the determination of chlorides, sulphates, lead, magnesium and iron have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Reference clause has been incorporated. Further, Packing and Marking clause has been updated.

Calcium chloride, technical, is used by the refrigeration and ice manufacturing industries for making brine and in the chemical industry as a raw material. It is also used for the purpose of spraying on road surfaces as an accelerative for concrete and in curing of concrete. Magnesium compounds are undesirable as refrigerating brines due to their sludge-forming properties, hence in the first revision more stringent limits for magnesium were specified. Anhydrous calcium chloride, technical, is used as a dehydrating agent in laboratory and a number of industries.

The composition of the Committee responsible for formulation of this Standard is given in [Annex C](#).

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.

Indian Standard
CALCIUM CHLORIDE — SPECIFICATION
(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for calcium chloride.

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 edition of these standards:

<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater:
(Part 2) : 2019/ ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (<i>first revision</i>)
(Part 65) : 2022/ ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (<i>first revision</i>)

3 TYPES AND GRADES**3.1 Types**

There shall be three types of the material, namely:

- a) *Type A* — Hydrated calcium chloride
(see [3.2](#));

- b) *Type B* — Anhydrous calcium chloride suitable as a drying agent in analytical work and in various industries; and
- c) *Type C* — Fused calcium chloride suitable for use in cement, paint and as a desiccant.

3.2 Grades

Type A of the material, that is, hydrated calcium chloride shall be of the following grades:

- a) *Grade 1* — Suitable as analytical reagent;
- b) *Grade 2* — Lye suitable for refrigeration and chemical industry; and
- c) *Grade 3* — Suitable for use as refrigerant in ice manufacture and as raw material in chemical industries also suitable for use for road construction purposes, for acceleration and curing of concrete.

4 REQUIREMENTS**4.1 Description**

The material shall be in the form of flakes or lumps, white in colour and free from extraneous impurities. All forms are deliquescent.

4.1.1 *Type A* — Grade 1 material may also be in the form of pellets, granules or sticks.

4.2 Size of Lumps

The size of lumps for Type B and Type C material shall be as agreed to between the purchaser and the supplier.

4.3 The material shall also comply with the requirements given in [Table 1](#) when tested in accordance with the methods prescribed in [Annex A](#). References to the relevant clauses of [Annex A](#) are given in col (8) of [Table 1](#).

4.4 Rejection

The material shall be rejected if it becomes caked or sticks to the containers before opening except for Type A, Grade 3.

5 PACKING AND MARKING**5.1 Packing**

5.1.1 Type B and Type C of the material shall be

To access Indian Standards click on the link below:

https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/knownyourstandards/Indian_standards/isdetails/

packed in flakes or lumps in moisture-proof bags or steel drums.

5.1.2 Type A, Grade 1 of the material shall be packed in glass container provided with suitable lids to keep away moisture.

5.1.3 Type A, Grade 2 of the material shall be packed in glass/polyethylene, carbuoys or shall be transported in tanker.

5.1.4 Type A, Grade 3 shall be packed in moisture-proof container as agreed to between the purchaser and the supplier.

5.2 Marking

The containers shall be securely closed and legibly and indelibly marked with the following information:

- a) Name, type and grade of the material;

- b) Name of the manufacturer and his trade-mark, if any;

- c) Net mass of the material; and

- d) Lot number in code or otherwise.

5.2.1 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 there under, and the products may be marked with the Standard Mark.

6 SAMPLING

The methods for drawing representative samples of the material and for determination of their conformity with the requirements of this standard shall be as given in [Annex B](#).

Table 1 Requirements for Calcium Chloride

(Clauses [4.3](#), [A-7.4](#), [A-8.4](#), [A-10.5](#), [A-11.4](#), [A-12.1.4](#), [A-13.1.4](#), [A-14.4](#), [A-15.2.3.3](#), [A-16.6](#), [A-17.5](#), [B-4.2](#) and [B-5.2](#))

SI No.	Characteristic	Requirement					Method of Test, to Ref
		Type A			Type B	Type C	
		Grade 1	Grade 2	Grade 3			
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Matter insoluble in water, percent by mass, <i>Max</i>	0.01	0.3	0.4	0.05	0.4	A-2
ii)	Calcium chloride (as CaCl ₂), percent by mass, <i>Min</i>	74.0	35.0	70.0	96.0	75.0	A-3
iii)	Magnesium chloride (as MgCl ₂), percent by mass, <i>Max</i>	—	0.5	0.7	0.5	0.7	A-4
iv)	Alkalinity [(as Ca (OH) ₂], percent by mass, <i>Max</i>	—	0.2	0.3	0.02	0.3	A-5
v)	Chlorides (as NaCl), percent by mass, <i>Max</i>	—	0.6	1.6	1.6	1.6	A-6 or A-20
vi)	Magnesium and alkali salts (as sulphates), percent by mass, <i>Max</i>	—	—	—	0.5	—	A-7

Table 1 (Concluded)

SI No.	Characteristic	Requirement					Method of Test, to Ref
		Type A			Type B	Type C	
		Grade 1	Grade 2	Grade 3	(6)	(7)	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
vii)	Oxidizing substances (as NO ₃), percent by mass, <i>Max</i>	0.003	—	—	—	—	A-8
viii)	Sulphates (as SO ₄), percent by mass, <i>Max</i>	0.010	—	—	0.020	—	A-9 or A-20
ix)	Ammonium salts (as NH ₄), percent by mass, <i>Max</i>	0.005	—	—	—	—	A-10
x)	Barium compounds (as Ba), percent by mass, <i>Max</i>	0.005	—	—	—	—	A-11
xi)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.005	—	—	0.005	—	A-12 or A-19
xii)	Iron (as Fe), percent by mass, <i>Max</i>	0.001	—	—	0.001	—	A-13 or A-19
xiii)	Magnesium (as Mg), percent by mass, <i>Max</i>	0.005	—	—	—	—	A-14 or A-19
xiv)	Potassium compounds (as K), percent by mass, <i>Max</i>	0.01	—	—	—	—	A-15
xv)	Sodium salts (as Na) percent by mass, <i>Max</i>	0.02	—	—	—	—	A-16
xvi)	Strontium salts (as Sr), percent by mass, <i>Max</i>	0.1	—	—	—	—	A-17
xvii)	pH of a 5 percent solution at 25 °C	4.5 to 8.5	7 to 8.5	7 to 8.5	7 to 8.5	7 to 8.5	A-18

ANNEX A

(Clause 4.3 and Table 1)

METHODS OF TEST FOR CALCIUM CHLORIDE

A-1 QUALITY OF REAGENTS

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

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

A-2 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-2.1 Outline of the Method

A known amount of the sample is dissolved in water and any insoluble matter present is filtered. The residue is washed and dried to constant mass.

A-2.2 Procedure

Transfer quickly about 20 g of the material from the bottle to a tared glass-stoppered weighing bottle, stopper and weigh. Transfer the weighed material to a breaker and dissolve in about 250 ml of water. Filter the solution through a tared gooch or sintered glass crucible (G No. 4) and wash the residue free from soluble salts. Collect the filtrate and washings in a 500 ml graduated flask and make up the volume with water. Use this solution for tests prescribed in [A-6](#) and [A-15.3](#).

Dry the crucible along with the residue to constant mass at a temperature of 105 °C to 110 °C.

A-2.3 Calculation

Matter insoluble in water, percent by mass

$$= \frac{100 M_1}{M_2}$$

where

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

M_2 = mass, in g, of the material taken for the test.

A-3 DETERMINATION OF CALCIUM CHLORIDE

A-3.1 Outline of the Method

The solution containing a known amount of the sample is titrated against EDTA solution using calcein as indicator.

A-3.2 Reagents

A-3.2.1 Standard Disodium Ethylene Diamine Tetra Acetate (EDTA) Solution

Weigh 3.72 g of disodium ethylene diamine tetra acetate dihydrate, dissolve in water and dilute in a graduated flask to 1 litre. Standardize the solution against the standard calcium chloride solution according to the procedure given in [A-3.3](#).

A-3.2.2 Standard Calcium Chloride Solution

Dissolve 1 g of calcium carbonate, dried at 120 °C to constant mass in the minimum quantity of concentrated hydrochloric acid (*see* IS 265) and then make up the volume to 1 litre. One millilitre of this solution is equivalent to 0.000 4 g of calcium.

A-3.2.3 Eriochrome Black T Indicator

Dissolve 0.1 g of the material in 20 ml of rectified spirit. This solution shall be prepared fresh every week.

A-3.2.4 Calcein Indicator — Mix 0.01 g of calcein thoroughly with 1 g of potassium chloride.

A-3.2.5 Buffer Solution

Dissolve 67.5 g of ammonium chloride in a mixture of 520 ml of ammonium hydroxide (sp gr 0.90) and 250 ml of water. Dissolve a mixture of 0.931 g of EDTA and 0.616 g of magnesium sulphate heptahydrate ($MgSO_4 \cdot 7H_2O$) in 50 ml of water, combine both the solutions and make up to 1 litre. Five millilitres of buffer solution added to 50 ml of water should not consume more than one drop of EDTA solution to change to distinct blue with eriochrome black T indicator.

A-3.2.6 Standard Sodium Hydroxide Solution — 2.5 N

A-3.3 Standardization of EDTA Solution

Standardize as given below:

a) *With Calcein Indicator*

Pipette out 50 ml of standard calcium chloride solution (*see* Note) in a 250 ml conical flask and add to it one millilitre of standard sodium hydroxide solution to adjust the pH of the solution to about 12. Add 10 mg of calcein indicator and titrate the solution against EDTA solution until the colour changes from yellow-green fluorescence to brown; and

b) *With Eriochrome Black T Indicator*

Pipette out 50 ml of standard calcium chloride solution (*see* Note) in 250 ml

conical flask and add 5 ml of buffer solution to adjust the pH of the solution to approximately 10. Add 5 drops of eriochrome black T indicator and titrate against standard EDTA solution until the wine-red colour changes to pure blue. (The volume of the solution to be titrated against standard EDTA solution should be approximately 50 ml and the strength of the standard EDTA solution should be adjusted in such a way that a similar volume of it is required for the complete titration).

NOTE — The aliquot for titration shall be neutral so that by adding standard sodium hydroxide solution or buffer solution the desired pH could be adjusted.

A-3.4 Prepared Sample Solution

Dissolve about 10 g of the material, accurately weighed in water and make up the volume to 1 000 ml.

A-3.5 Procedure

Pipette out 10 ml of the prepared sample solution (see [A-3.4](#)) in a 250 ml conical flask and dilute with 40 ml of water. Add 1.5 ml of standard sodium hydroxide solution followed by 10 mg of calcein indicator. Titrate against the standard EDTA solution till the colour changes from yellow-green fluorescence to brown.

A-3.6 Calculation

Calcium (as Ca), percent by mass

$$= \frac{10\,000 V_1 A_1}{M}$$

where

V_1 = volume, in ml, of standard EDTA solution used;

A_1 = mass, in g, of calcium equivalent to one millilitre of standard EDTA solution as obtained in [A-3.3\(a\)](#); and

M = mass, in g, of the material taken in [A-3.4](#).

A-3.6.1 Calcium Chloride (as CaCl_2)

Deduct the calcium corresponding to calcium hydroxide (see [A-5.4](#)) from the total calcium present and express the balance of calcium as percent calcium chloride (as CaCl_2) by multiplying with 2.766.

A-4 DETERMINATION OF MAGNESIUM CHLORIDE

A-4.1 Outline of the Method

The solution containing a known amount of the sample is titrated against EDTA solution using eriochrome Black T as indicator.

A-4.2 Reagents

A-4.2.1 Buffer Solution — same as given in [A-3.2.5](#)

A-4.2.2 Eriochrome Black T Indicator — same as given in [A-3.2.3](#)

A-4.2.3 Standard Disodium Ethylene Diamine Tetra Acetate (EDTA) Solution — same as given in [A-3.2.1](#).

A-4.3 Procedure

Pipette out 10 ml of the prepared sample solution (see [A-3.4](#)) in a 250 ml conical flask and dilute with 35 ml of water. Add 5 ml of buffer solution and 5 drops of eriochrome black T indicator. Titrate against standard EDTA solution till the wine-red colour of the solution changes to blue. Note the reading of the burette.

A-4.4 Calculation

Magnesium, percent by mass

$$= \frac{6.086 (V_2 A_2 - V_1 A_1)}{M}$$

where

V_2 = volume, in ml, of standard EDTA solution used in [A-4.3](#);

A_2 = mass, in g, of calcium equivalent to 1 ml of standard EDTA solution as obtained in [A-3.3\(a\)](#);

V_1 = volume, in ml, of standard EDTA solution used in [A-3.5](#);

A_1 = mass, in g, of calcium equivalent to 1 ml of standard EDTA solution as obtained in [A-3.3\(b\)](#); and

M = mass, in g, of the material taken in [A-3.4](#).

A-4.5 Magnesium Chloride (as MgCl_2)

Express magnesium present as magnesium chloride (as MgCl_2) by multiplying with 3.916.

A-5 DETERMINATION OF ALKALINITY

A-5.1 Outline of the Method

A known amount of the sample is dissolved in freshly boiled and cooled water, filtered and titrated against hydrochloric using phenolphthalein as indicator.

A-5.2 Reagents

A-5.2.1 Standard Hydrochloric Acid — 0.1 N

A-5.2.2 Phenolphthalein Indicator

Dissolve 0.1 g of phenolphthalein in 100 ml of rectified spirit.

A-5.3 Procedure

Weigh accurately about 10 g of the sample, dissolve it in 50 ml of freshly boiled and cooled water, filter the solution through folded filter paper, wash the filter paper thoroughly but as quickly as possible and collect the filtrate and washings in a 200 ml conical flask. Titrate the collected solution against standard hydrochloric acid using phenolphthalein as indicator.

A-5.4 Calculation

$$\begin{aligned} \text{Alkalinity [as Ca(OH)}_2\text{] percent by mass} \\ = \frac{3.705 VN}{M} \end{aligned}$$

where

V = volume, in ml, of the standard hydrochloric acid used for the titration;

N = normality, of standard hydrochloric acid; and

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

A-6 DETERMINATION OF ALKALI CHLORIDES**A-6.1 Method A****A-6.1.1 Outline of the Method**

An aliquot of the solution of the sample obtained after determination of matter insoluble in water is treated with dilute nitric acid and excess of silver nitrate. The precipitate is filtered and washed. The filtrate together with the washings is titrated with standard thiocyanate solution using ferric ammonium sulphate as indicator.

A-6.1.2 Reagents

A-6.1.2.1 Dilute nitric acid — approximately 5 N

A-6.1.2.2 Standard silver nitrate solution — 0.1 N

A-6.1.2.3 Standard ammonium or potassium thiocyanate solution — 0.1 N

A-6.1.2.4 Ferric ammonium sulphate solution — approximately 40 percent (m/v) solution in water

A-6.1.3 Procedure

Transfer 50 ml of the solution reserved in [A-2.2](#) to a 250 ml graduated flask and make up the volume of solution with water. Pipette 50 ml of this solution to a breaker and dilute to 100 ml. Add 5 ml of dilute nitric acid and then add in excess a measured volume of silver nitrate solution. Stir to coagulate the precipitate. Filter, and wash the precipitate with

dilute nitric acid. Add to the combined filtrate and washings about 2 ml to 3 ml of ferric ammonium sulphate solution and titrate the excess of silver nitrate with standard ammonium or potassium thiocyanate solution.

A-6.1.3.1 Carry out a blank determination at the same time.

A-6.1.4 Calculation

$$\begin{aligned} \text{Chloride (as Cl), percent by mass} \\ = \frac{3.546 (V_1 - V_2)N}{M} \end{aligned}$$

where

V_1 = volume, in ml, of standard ammonium or potassium thiocyanate solution required for the blank;

V_2 = volume, in ml, of standard ammonium or potassium thiocyanate solution required for the test with the material;

N = normality, of standard ammonium or potassium thiocyanate solution; and

M = mass, in g, of the material in the aliquot.

A-6.1.5 Alkali Chlorides (as NaCl)

Deduct the chloride corresponding to calcium chloride and magnesium chloride from the total chloride present and calculate the residue chloride (as NaCl) by multiplying with 1.648 7.

A-6.2 Alternative Method

Chlorides may alternatively be determined by ICP-OES instrumental test method as prescribed at [A-20](#).

A-7 TEST FOR MAGNESIUM AND ALKALI SALTS**A-7.1 Outline of the Method**

The sample is dissolved in water, acidified and heated to boiling. A slight excess of hot ammonium oxalate is added and rendered alkaline with ammonium hydroxide. Cooled and filtered. Filtrate is acidified with sulphuric acid and nitric acid and evaporated to dryness. The residue is dissolved in water and filtered. The combined filtrate and washings are evaporated to dryness and finally ignited to constant mass.

A-7.2 Reagents

A-7.2.1 Hydrochloric Acid — 20 percent (v/v)

A-7.2.2 Methyl Red Indicator Solution

Dissolve 0.15 g of methyl red in 500 ml of water

A-7.2.3 Ammonium Oxalate Solution

Dissolve 35 g of ammonium oxalate [(NH₄)₂C₂O₄.H₂O] in water and make up to 1 000 ml

A-7.2.4 Ammonium Hydroxide Solution — 10 percent (v/v)

A-7.2.5 Sulphuric Acid — 10 percent (v/v)

A-7.2.6 Concentrated Nitric acid — (see IS 264)

A-7.3 Procedure

Dissolve 1 g of the material in 100 ml of water, add 5 ml of hydrochloric acid and few drops of methyl red indicator solution, and heat to boiling. Add dropwise with stirring 100 ml of warm ammonium oxalate solution. Heat the solution to 70 °C to 80 °C, and add dropwise ammonium hydroxide solution, until it is just alkaline to methyl red. Dilute to 250 ml and allow to cool for 1 h. Filter, add to 125 ml of the filtrate 5 ml of sulphuric acid, evaporate to about 30 ml, and cool. Add 25 ml of nitric acid and evaporate to dryness on a steam-bath. Dissolve the residue in a small quantity of water, filter through a small filter paper, and wash. Transfer the filtrate and washings to a small tared porcelain basin. Evaporate to dryness and ignite gently to volatilize the excess acids and salts and finally ignite at (800 ± 25) °C for 25 min. Weigh the tared dish.

A-7.4 The limit specified in [Table 1](#) shall be deemed as not having been exceeded if the ignited residue does not weigh more-than 0.01 g.

A-8 TEST FOR OXIDIZING SUBSTANCES**A-8.1 Outline of the Method**

The material is dissolved in ice cold sulphuric acid. Diphenylamine is added and digested on a steam-bath. The colour produced is compared with the colour produced in a control test.

A-8.2 Reagents

A-8.2.1 Concentrated Sulphuric Acid — see IS 266

A-8.2.2 Diphenylamine Solution

Dissolve 10 mg of colourless diphenylamine in 100 ml of sulphuric acid. In another beaker dissolve 2 g of ammonium chloride in 200 ml of water. Cool both solutions in ice-bath. Cautiously add the sulphuric acid solution to the water solution, taking care to keep the resulting solution cold.

A-8.2.3 Standard Nitrate Solution

Dissolve 0.164 g of potassium nitrate (KNO₃) in water and dilute to 100 ml. Dilute 10 ml of this

solution to 1 000 ml. One millilitre of the solution contains 0.01 mg of nitrate (as NO₃).

A-8.2.4 Sodium Carbonate**A-8.3 Procedure**

Transfer 0.2 g of the sample into a dry beaker. Cool it thoroughly in an ice-bath and add 22 ml of sulphuric acid, already cooled to ice-bath temperature. Allow the mixture to warm to room temperature and swirl the beaker at intervals to effect quiet solution with gentle evolution of hydrogen chloride. When solution is complete, add 3 ml of diphenylamine solution and digest on steam-bath for 90 min. In another dry beaker evaporate to dryness a solution containing 0.6 ml of the standard nitrate solution and 0.01 g of sodium carbonate. Treat the residue in the same way as the material.

A-8.4 The limit prescribed in [Table 1](#) shall be taken as not having been exceeded if the colour produced with the material is not darker than that produced with the standard nitrate solution.

A-9 DETERMINATION OF SULPHATES**A-9.1 Method A****A-9.1.1 Outline of the Method**

The solution of the sample which is weakly acid medium is heated to boil for 5 min and treated with hot barium chloride solution. The barium sulphate precipitate filtered, washed and ignited to constant mass.

A-9.1.2 Reagents

A-9.1.2.1 Methyl Red Indicator Solution — same as in [A-7.2.2](#)

A-9.1.2.2 Ammonium Hydroxide Solution — free from carbonate

A-9.1.2.3 Concentrated Hydrochloric Acid — see IS 265

A-9.1.2.4 Barium Chloride Solution

Dissolve 120 g of barium chloride (BaCl₂.2H₂O) in water and dilute to 1 litre

A-9.1.3 Procedure

Dissolve 10 g of the material in 100 ml of water and heat to boiling. Add a few drops of methyl red indicator solution, make alkaline with ammonium hydroxide solution and boil for 5 min. Filter through a small filter paper, and wash with a little hot water. Reject the residue. Neutralize the filtrate with hydrochloric acid and add 1 ml in excess. Determine the sulphate content as prescribed in IS 2317.

A-9.1.4 Calculation

Sulphates (as SO₄), percent by mass

$$= 41.15 \frac{M_1}{M_2}$$

where

M_1 = mass, in g, of the precipitate; and

M_2 = mass, in g, of the material contained in the solution taken for precipitation.

A-9.2 Alternative Method

Sulphates may alternatively be determined by ICP-OES instrumental test method as prescribed at [A-20](#).

A-10 TEST FOR AMMONIUM SALTS**A-10.1 Outline of the Method**

The solution of the sample is treated with sodium hydroxide and filtered. To the filtrate, Nessler solution is added and the colour produced is compared with that produced in a control test containing definite amount of ammonium hydroxide.

A-10.2 Reagents

A-10.2.1 Sodium Hydroxide Solution — 10 percent (m/v)

A-10.2.2 Nessler Solution

Dissolve 143 g of sodium hydroxide in 700 ml of water. Dissolve 50 g of red mercuric iodide and 40 g of potassium iodide in 200 ml of water. Pour the iodide solution into hydroxide solution and dilute to 1 000 ml. Allow to settle and use the clear supernatant liquid.

A-10.2.3 Standard Ammonium Chloride Solution

Dissolve 0.296 g of ammonium chloride (NH₄Cl) in water and dilute to 100 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.01 mg of ammonium (as NH₄).

A-10.3 Preparation of Test Solution

Dissolve 50 g of the material in about 200 ml of water, filter carefully, if necessary, and dilute to 250 ml in a measuring flask (Solution A).

A-10.4 Procedure

Dilute 10 ml of the Solution A ([A-10.3](#)) to 45 ml and add 15 ml of sodium hydroxide solution. Filter through a sintered glass crucible, previously washed with sodium hydroxide solution. Take 6 ml of the filtrate, dilute with water to 50 ml and add 2 ml of

Nessler solution. In a control test, having an equal volume of the solution and containing 1 ml of standard ammonium chloride solution and 1.5 ml of sodium hydroxide, add 2 ml of Nessler solution.

A-10.5 The limit prescribed in [Table 1](#) shall be taken as not having been exceeded if the colour produced with the material is not darker than that provided with the standard solution.

A-11 TEST FOR BARIUM SALTS**A-11.1 Outline of the Method**

The solution of the sample is treated with sodium acetate, acetic and potassium dichromate. The turbidity due to barium chromate produced is compared with that produced in a control test.

A-11.2 Reagents

A-11.2.1 Sodium Acetate — solid

A-11.2.2 Acetic Acid

A-11.2.3 Standard Barium Solution

Dissolve 0.178 g of barium chloride (BaCl₂·H₂O) in water and dilute to 1 000 ml.

A-11.2.4 Potassium Dichromate Solution

Dissolve 10 g of potassium dichromate (K₂Cr₂O₇) in water and dilute to 100 ml with water.

A-11.3 Procedure

Take in a tall beaker 15 ml of Solution A ([A-10.3](#)) and add to it 2 g of sodium acetate and 0.05 ml of acetic acid. Add 2 ml of potassium dichromate solution and allow to stand for 15 min.

In another tall beaker take 5 ml of standard barium solution, and add to it 2 g of sodium acetate, 0.05 ml of acetic acid and dilute to 15 ml. Add 2 ml of potassium dichromate solution, and allow to stand for 15 min.

A-11.4 The limit prescribed in [Table 1](#) shall be taken as not having been exceeded if the turbidity produced with the material is not greater than that produced in the control test.

A-12 TEST FOR HEAVY METALS**A-12.1 Method A****A-12.1.1 Outline of the Method**

The solution of the sample in weakly acetic acid medium is treated with hydrogen sulphide water and the colour produced is compared with that produced with a standard lead solution.

A-12.1.2 Reagents**A-12.1.2.1 Acetic Acid** — 1 N**A-12.1.2.2 Standard lead solution**

Dissolve 0.160 g of lead nitrate [Pb(NO₃)₂] in 100 ml of dilute nitric acid (1 : 99) and dilute to 1 000 ml. Take 20 ml of this solution and dilute to 100 ml with water. One millilitre of this diluted solution contains 0.2 mg of lead (as Pb).

A-12.1.2.3 Hydrogen sulphide water

Saturate water with hydrogen sulphide. This solution shall be freshly prepared.

A-12.1.3 Procedure

Take 30 ml of Solution A ([A-10.3](#)) in a Nessler cylinder B, add 1 ml of acetic acid and dilute to 40 ml. In another Nessler cylinder C take 1 ml of standard lead solution, add to it 10 ml of Solution A ([A-10.3](#)) and 1 ml of acetic acid, dilute to 40 ml. Add 10 ml of hydrogen sulphide water to each. Dilute the contents of each cylinder to 50 ml and mix well.

A-12.1.4 The limits prescribed in [Table 1](#) shall be taken as not having been exceeded if the colour produced in cylinder B is not more intense than that in cylinder C.

A-12.2 Alternative Method

Lead may alternatively be determined by ICP-OES instrumental test method as prescribed at [A-19](#) or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-13 TEST FOR IRON**A-13.1 Method A****A-13.1.1 Outline of the Method**

The solution of the sample is oxidized with ammonium persulphate and treated with ammonium thiocyanate. The colour produced is compared with that produced with a standard iron solution.

A-13.1.2 Reagents

A-13.1.2.1 Dilute hydrochloric acid — approximately 5 N

A-13.1.2.2 Dilute sulphuric acid — approximately 5 N

A-13.1.2.3 Ammonium persulphate — solid

A-13.1.2.4 Butanolic potassium thiocyanate solution

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient N-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-13.1.2.5 Standard iron solution

Dissolve 0.702 g of ferrous ammonium sulphate [Fe(NH₄)₂ (SO₄)₂.6H₂O] in 10 ml of 10 percent sulphuric acid and dilute to 100 ml with water. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the solution contains 0.01 mg of iron (as Fe).

A-13.1.3 Procedure

Take 10 ml of the solution A ([A-10.2](#)) and dilute with 20 ml of water and transfer to a Nessler cylinder. Add 5 ml of hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Dilute to the mark with water. Shake vigorously for 30 s and allow the liquids to separate. Carry out a control test in the other Nessler cylinder with 1 ml of standard solution.

A-13.1.4 The limits prescribed in [Table 1](#) shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-13.2 Spectroscopic Method (Method B)**A-13.2.1 General**

Trivalent iron is reduced by means of hydroxyl-ammonium chloride and a bivalent iron-2, 2-bipyridyl complex at pH 3.1, at a temperature of 75 °C is formed. Photometric measurement of the coloured complex is done at a wavelength of about 520 nm.

A-13.2.2 Apparatus

A-13.2.2.1 pH Meter — fitted with glass electrode

A-13.2.2.2 Spectrophotometer — provided with a 1 cm cell

A-13.2.3 Reagents

A-13.2.3.1 Concentrated hydrochloric acid — see IS 265

A-13.2.3.2 2,2-Bipyridyl Solution

Dissolve 0.50 g of 2,2-bipyridyl in 10 ml of concentrated hydrochloric acid and dilute to 100 ml with water.

A-13.2.3.3 Ammonium acetate solution

Dissolve 300 g of ammonium acetate in water and make the volume to 1 000 ml.

A-13.2.3.4 Hydroxyl ammonium chloride

Dissolve 10 g of hydroxyl-ammonium chloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) in water and dilute to 100 ml.

A-13.2.3.5 Standard iron solution

Dissolve 0.702 g of ferrous ammonium sulphate [$\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2\cdot 6\text{H}_2\text{O}$] in 20 ml of 10 percent sulphuric acid and dilute with water to exactly 500 ml. Further dilute 100 ml of this solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.02 mg of iron (as Fe).

A-13.2.4 Procedure (for Technical and Reagent Grade)**A-13.2.4.1 Preparation of sample solution**

Weigh 50 g of the material to the nearest 0.01 g in a small glass beaker. Transfer quantitatively to a 500 ml volumetric flask and make up to the mark with carbon dioxide-free water. Mix and use the sample solution for tests. Pipette out 5 ml of the sample solution in a 100 ml volumetric flask and dilute to the mark. Shake the contents. This diluted solution shall be called the diluted sample solution.

A-13.2.4.2 Blank test

At the same time as the analysis, carry out a blank test using the procedure described in [A-13.2.4.4](#) and the same quantities of all reagents.

A-13.2.4.3 Preparation of calibration curve —
Proceed the method as prescribed below:a) *Preliminary check of pH*

Place 5 ml of concentrated hydrochloric acid in a beaker of suitable capacity (100 ml for example), dilute to approximately 50 ml, add 1 ml of hydroxylammonium chloride solution and 5 ml of 2,2 - bipyridyl solution. Allow to stand for about 10 min and using the pH meter, adjust the pH of the solution to 3.1 by addition of ammonium acetate solution. Note the quantity of ammonium acetate added for the pH adjustment and then discard the solution.

b) *Preparation of standard solutions*

Into each of a series of five beakers of suitable capacity (100 ml, for example),

place the quantities of standard iron solution indicated below:

<i>Sl No.</i>	<i>Standard Iron Solution (ml)</i>	<i>Corresponding to Iron (Fe) (μg)</i>
(1)	(2)	(3)
i)	*0	0
ii)	5.0	100 ($5.0 \times 0.02 \text{ mg} = 0.1 \text{ mg}$)
iii)	10.0	200 ($10.0 \times 0.02 \text{ mg} = 0.2 \text{ mg}$)
iv)	15.0	300 ($15.0 \times 0.02 \text{ mg} = 0.3 \text{ mg}$)
v)	25.0	500 ($25.0 \times 0.02 \text{ mg} = 0.5 \text{ mg}$)

Then add 5 ml of concentrated hydrochloric acid and dilute to approximately 50 ml.

c) *Colour development*

To each of the above-mentioned solutions, add 1 ml of hydroxyl ammonium chloride solution and 5 ml of 2,2-bipyridyl solution. Allow to stand for about 10 min, then add the quantity of ammonium acetate solution for adjusting pH of 3.1. Heat the solutions on a water-bath at approximately 75 °C for about 15 min and then cool to room temperature. Transfer to 100 ml one-mark volumetric flasks, dilute to the mark and mix thoroughly.

d) *Photometric measurement*

Measure the optical density of each solution using the spectrophotometer at a wavelength of about 520 nm, adjusting the instrument to zero optical density using as reference the compensation solution.

e) *Preparation of calibration graph*

Prepare a calibration graph for iron (Fe) content in micrograms per 100 ml of the standard solution as abscissa and the corresponding values of optical density as ordinate.

A-13.2.4.4 Determination

Pipette out 10 ml of the diluted sample solution (see [A-13.2.4.1](#)) into a beaker of suitable capacity (100 ml) and dilute to approximately 50 ml.

*Compensation solution

Add 1 ml of hydroxylammonium chloride solution and 5 ml of 2,2 - bipyridyl solution. Allow to stand for about 10 min, then using the pH meter, adjust the pH of the solution to 3.1 by addition of ammonium acetate solution. Heat the solution on a water-bath to approximately 75 °C for about 15 min and cool to room temperature. Transfer the solution quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix thoroughly. Measure the optical density of the solution adjusting the instrument to zero optical density with the blank test solution and following the procedure described in [A-13.2.4.3\(d\)](#).

A-13.2.4.5 Calculation

By reference to the calibration graph, read the iron content corresponding to the photometric measurement of the sample and calculate as given below:

$$\begin{aligned} & \text{Iron (as Fe), percent by mass} \\ & = \frac{A \times 100 \times 100}{E} \end{aligned}$$

where

A = mass, in g, of iron determined in the sample solution; and

E = mass, in g, of the sample taken for the test in [A-13.2.4.1](#)

A-13.3 Alternative Method

Iron may alternatively be determined by ICP-OES instrumental test method as prescribed at [A-19](#) or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-14 TEST FOR MAGNESIUM

A-14.1 Outline of the Method

The solution of the sample is treated with titan yellow solution and sodium hydroxide. The colour developed is compared with that produced with a standard magnesium solution.

A-14.2 Reagents

A-14.2.1 Sodium Hydroxide Solution — approximately 1 N

A-14.2.2 Titan Yellow Solution — 0.1 percent solution in water

A-14.2.3 Standard Magnesium Solution

Dissolve 1.014 g of clear crystals of magnesium sulphate heptahydrate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in water and

dilute to 100 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of this diluted solution is equivalent to 0.01 mg of magnesium (as Mg)

A-14.3 Procedure

Take in a Nessler cylinder B 7.5 ml of Solution A ([A-10.3](#)) and dilute to 20 ml. Add to it 0.15 ml of the titan yellow solution and then 2 ml of sodium hydroxide solution. Mix well and allow to stand for 10 min after which dilute to 50 ml and mix well. In another Nessler cylinder C take 2.5 ml of Solution A ([A-10.3](#)) and add to it 5 ml of standard magnesium solution and dilute to 20 ml. Treat it also in the same way as described above. Compare the colour developed in both the cylinders.

A-14.4 The limit prescribed in [Table 1](#) shall be taken as not having been exceeded if the colour in cylinder B is not darker than that in cylinder C.

A-14.5 Alternative Method

Magnesium may alternatively be determined by ICP-OES instrumental test method as prescribed at [A-19](#) or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

A-15 DETERMINATION OF POTASSIUM

A-15.1 Outline of the Method

Potassium is determined instrumentally by the use of a flame photometer. Alternatively the solution of the sample at ice cold temperature is treated with sodium tetra phenyl boron, the precipitate is filtered, washed and dried at 110 °C.

A-15.2 Flame Photometer Method

A-15.2.1 Apparatus

A-15.2.1.1 Flame photometer — with red-sensitive photo-tube

A-15.2.1.2 Oxy-hydrogen burner

A-15.2.2 Reagents

A-15.2.2.1 Concentrated hydrochloric acid — see IS 265

A-15.2.2.2 Standard potassium chloride solution

Dissolve 0.191 g of potassium chloride (KCl) in water and dilute to 1 000 ml. Take 100 ml of this solution and dilute to 1 000 ml with water. One millilitre of this diluted solution is equivalent to 0.01 mg of potassium (as K).

A-15.2.3 Procedure**A-15.2.3.1 Preparation of solutions****Sample Solution S**

To a 5 ml aliquot of Solution A (**A-10.3**), add 1 ml of hydrochloric acid and dilute to 100 ml with water.

Control Solution C

To another 5 ml aliquot of Solution A (**A-10.3**), add 1 ml of standard potassium chloride solution, 1 ml of hydrochloric acid and dilute to 100 ml with water.

A-15.2.3.2 Set the flame photometer according to the directions of the manufacturer of the apparatus. Observe the maximum intensity of radiation produced by control Solution C at 767 nm (potassium line). Observe the intensity of radiation produced by sample Solution S at 767 nm and also at 750 nm with the same adjustments of the instrument.

A-15.2.3.3 The limit prescribed in [Table 1](#) shall be taken as not having been exceeded if the difference (D_1) between the intensities observed at 767 nm and 750 nm for sample Solution S does not exceed the difference (D_2) between the intensities observed at 767 nm for control Solution C and sample Solution S.

A-15.3 Sodium Tetra Phenyl Boron Method**A-15.3.1 Apparatus**

A-15.3.1.1 Sintered glass crucible — G No. 4 or Gooch crucible

A-15.3.1.2 Ice-bath**A-15.3.2 Reagents****A-15.3.2.1 Sodium tetra phenyl boron**

Dissolve 1 g of sodium tetra phenyl boron in 100 ml of 0.01 N sodium hydroxide solution. Before use cool the solution in ice.

A-15.3.2.2 Wash solution

Recrystallize potassium tetra phenyl boron from acetone and saturate water with the recrystallized product. The solution shall be prepared fresh every day.

A-15.3.3 Procedure

Transfer 25 ml of the solution reserved in [A-2.2](#) into a 100 ml conical flask. Place the flask in an ice-bath for 5 to 10 min. When the solution has cooled, add 25 ml of sodium tetra phenyl boron solution previously cooled in ice. Mix by gentle swirling and leave in the ice-bath for 5 min to 10 min. Filter through

a sintered glass crucible (G No. 4) or a Gooch crucible, wash the flask and the precipitate with three 10 ml portions of the wash solution and dry the crucible containing the precipitate for one hour at 110 °C to 120 °C. Cool in a desiccator and weigh.

A-15.3.4 Calculation

Potassium (as K), percent by mass (on dry mass)

$$= \frac{21\,820\,M_1}{M_2 \times 9(100 - M)}$$

where

M_1 = mass, in g, of the precipitate;

M_2 = mass, in g, of the prepared in sample taken for the test in [A-2.2](#); and

M = percent, moisture content.

A-16 TEST FOR SODIUM**A-16.1 Outline of the Method**

Sodium is determined instrumentally by the use of a flame photometer

A-16.2 Apparatus

A-16.2.1 Flame Photometer — same as in [A-15.2.1.1](#)

A-16.2.2 Oxy-Hydrogen Burner — same as in [A-15.2.1.2](#)

A-16.3 Reagent

A-16.3.1 Concentrated Hydrochloric Acid — see IS 265

A-16.3.2 Standard Sodium Chloride Solution

Dissolve 0.254 g of sodium chloride (NaCl), dried at (105 ± 5) °C in water and dilute to 1 000 ml. One millilitre of the solution contains 0.1 mg of sodium (as Na).

A-16.4 Procedure**A-16.4.1 Preparation of Solution****A-16.4.1.1 Sample Solution S**

To a 5 ml aliquot of Solution A (**A-10.3**), add 1 ml of hydrochloric acid and dilute to 100 ml with water.

A-16.4.1.2 Control Solution C

To another 5 ml aliquot of Solution A (**A-10.3**), add 2 ml of standard sodium chloride solution and 1 ml of hydrochloric acid and dilute to 100 ml with water.

A-16.5 Procedure

Set the flame photometer according to the directions of the manufacturer of the apparatus. Observe the

emission of control Solution C at 589 nm (sodium line) and the emission of sample Solution S at 589 nm and 580 nm.

A-16.6 The limit prescribed in [Table 1](#) shall be taken as not having been exceeded if the difference (D_1) between the intensity observed for sample Solution C at 580 nm does not exceed the difference (D_2) observed at 589 nm between sample Solution S and control Solution C.

A-17 TEST FOR STRONTIUM

A-17.1 Outline of the Method

Strontium is determined instrumentally by the use of a flame photometer.

A-17.2 Apparatus

A-17.2.1 Flame Photometer — same as in [A-15.2.1.1](#)

A-17.2.2 Oxy-Hydrogen Burner — same as in [A-15.2.1.2](#)

A-17.3 Reagents

A-17.3.1 Concentrated Hydrochloric Acid — see IS 265

A-17.3.2 Standard Strontium Nitrate Solution

Dissolve 0.242 g of strontium nitrate [$\text{Sr}(\text{NO}_3)_2$] in water and dilute to 100 ml. One millilitre of this solution contains 0.01 mg of strontium (as Sr).

A-17.4 Procedure

A-17.4.1 Preparation of Solutions

A-17.4.1.1 Sample Solution S

To a 5 ml aliquot of Solution A ([A-10.3](#)), add 1 ml of hydrochloric acid and dilute to 100 ml with water.

A-17.4.1.2 Control Solution C

To another 5 ml aliquot of Solution A ([A-10.3](#)), add 10 ml of standard strontium nitrate solution and 1 ml of hydrochloric acid, and dilute to 100 ml with water.

A-17.4.2 Set the flame photometer according to the directions of the manufacturer of the apparatus. Observe the emission of control Solution C at 460.7 nm (strontium line) and the emission of sample Solution S at 460.7 nm and a wave length of 2 nm or 4 nm less than 460.7 nm.

A-17.5 The limit prescribed in [Table 1](#) shall be taken as not having been exceeded if the difference (D_1) between the intensity observed for

sample Solution S at 460.7 nm and 460.7 nm, - 2 nm or - 4 nm does not exceed the difference (D_2) observed at 460.7 nm between the Solution S and Solution C.

A-18 DETERMINATION OF pH

A-18.1 Outline of the Method

pH is determined instrumentally by the use of a pH meter

A-18.2 Apparatus

A-18.2.1 pH Meter

Any suitable pH meter equipped with a standard calomel electrode and glass electrode. The meter shall be accurate to 0.05 pH unit in the pH range of 4.01 to 9.18.

A-18.3 Procedure

Dissolve 10 g of the material in 200 ml of water at 25 °C. Determine the pH in accordance with the directions provided by the manufacturer.

A-19 DETERMINATION OF IRON, LEAD AND MAGNESIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-19.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

A-19.2 Recommended Wavelength, Limit of Quantification and Important Spectral Interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in [Table 2](#). Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences*(Clauses [A-19.2](#) and [A-19.4](#))*

SI No.	Element	Wavelength (nm)	Approximately Achievable Limits		Interfering Elements
			Radial Viewing (μg)	Axial Viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
ii)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)	(20)	Cr, Fe
		217.00			
iii)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr

A-19.3 Reagents and Solutions**A-19.3.1. Nitric Acid (65 percent) Suprapure****A-19.3.2 Standard Stock Solution**

Either Prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1 000 $\mu\text{g}/\text{ml}$ of Lead or Iron in 2 percent to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-19.3.3. Standard Solution

Pipette out 5 ml from 100 $\mu\text{g}/\text{ml}$ standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 $\mu\text{g}/\text{ml}$ solution. From this 5 $\mu\text{g}/\text{ml}$ solution, an aliquot of 1.0 ml, 3.0 ml and 5.0 ml taken in 50 ml volumetric Flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1 $\mu\text{g}/\text{ml}$, 0.3 $\mu\text{g}/\text{ml}$ and 0.5 $\mu\text{g}/\text{ml}$ solution of respective elements under reference.

A-19.3.4 Sample Preparation

Weigh about 2.5 g sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

A-19.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis,

polytetrafluorethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

A-19.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from [Table 2](#).

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

A-19.5 Procedure**A-19.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions ([A-19.3.3](#)). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities.

Flush the system with the reagent blank solution between each standard.

A-19.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

Begin the sample run by flushing the system with the reagent blank solution ([A-19.3.5](#)) between each sample.

It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples.

Analyze the sample solution and calculate the concentration in $\mu\text{g/ml}$ of the lead (or Iron) in the sample solution.

NOTE — It is recommended that IS 3025(Part 2)/ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

A-19.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

- a) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot; and
- b) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (dilution factor).

A-20 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-20.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing

the pH of the column. Similarly in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

A-20.2 Equipment

A-20.2.1 Anion Guard Column — a protector of the separator column

A-20.2.2 Anion Separator Column — suitable for selective separation of ions under analysis

A-20.2.3 Anion Suppressor Device — anion micromembrane suppressor is used to analyse the data

A-20.2.4 Detector — conductivity detector

A-20.2.5 Software — software suitable for control of various operating parameters, receiving inputs and analysis of all data.

A-20.2.6 Sample loop of 100 μl , 200 μl , 500 μl or 1 000 μl be used to determine ionic concentration as per instrument manual and practice.

A-20.3 Reagents

A-20.3.1 Glass or polyethylene sample bottles

A-20.3.2 Distilled Water or Deionized Water free from the Anions of Interest

A-20.3.4 Eluent — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 litres of water.

A-20.3.5 Micromembrane Suppressor Solution (0.025 N of Sulphuric Acid)

Dilute 2.8 ml of concentrated sulphuric acid in 4 litres of water.

A-20.4 Standard Solutions

A-20.4.1 Chloride

Dissolve NaCl, 1.648 5 g in 1 litre of reagent water.

A-20.4.2 Sulphate

Dissolve 1.81 g of potassium sulphate in 1 litre of reagent water.

A-20.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1 ml to 1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-20.6 Procedure

Dissolve between 1 g to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an

appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used.

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 μ membrane. This membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-20.7 Data Analysis and Calculations

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/L or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

ANNEX B

(Clause 6)

SAMPLING OF CALCIUM CHLORIDE

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 In drawing, storing, preparing and handling test samples the following precautions shall be observed.

B-1.2 Samples shall not be taken at a place which is exposed to weather.

B-1.3 Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.5 The samples shall be placed in suitable dry and air-tight containers.

B-1.6 Each sample container shall be sealed air-tight after filling and shall be marked with full details of sampling, the date of sampling and the year of manufacture.

B-2 SCALE OF SAMPLING**B-2.1 Lot**

All the containers in a single consignment of calcium chloride of the same type and grade drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots. If the material is stored in large drums, each drum will constitute a lot.

B-2.2 The number of containers (n) to be selected from the lot shall depend upon the size of the lot (N) and shall be in accordance with [Table 3](#).

Table 3 Number of Containers to be Selected(Clause [B-2.2](#))

Sl No.	Lot Size	Number of Containers to be Selected
(1)	N (2)	n (3)
i)	4 to 50	3
ii)	51 to 100	4
iii)	101 to 150	5
iv)	151 to 300	7
v)	301 and above	10

NOTE — When the size of the lot is 3 or less, the number of containers to be selected and the criterion for judging conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

B-2.3 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them in-one order as 1, 2, 3, . . . up to r and so on, where r is the integral part of N/n (N and n being the lot size and sample size, respectively). Every r^{th} container thus counted shall be withdrawn to constitute the sample.

B-2.4 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of this specifications.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 From each of the n containers selected according to [B-2.3](#), a portion of the material, about 200 g, shall be drawn with the help of a suitable sampling instrument.

B-3.2 Out of these portions, equal quantities of the material shall be drawn and mixed thoroughly to form a composite sample of about 500 g. The composite sample shall be divided into three parts, one for the purchaser, and another for the supplier and the third used as a referee sample.

B-3.2.1 If the lot consists of a single drum, composite sample shall be formed by mixing the material drawn from a number of places in the drum with suitable sampling instrument.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.4 All the individual samples and the composite sample shall be transferred to separate sampling containers. All the containers shall be sealed and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier and shall be kept at a place agreed to

between the purchaser and the supplier, to be used in case of any dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 For all the different types and grades of the material, tests for the determination of calcium chloride content shall be performed on each of the individual samples.

B-4.2 Tests for the determination of all other characteristics given in [Table 1](#) shall be performed on each of the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

From the test results, the average (X) for the calcium chloride content and the range (R) shall be computed (range R being defined as the difference between the

maximum and the minimum values of the test results). The appropriate expression as shown in col (6) of [Table 4](#) shall be calculated for the characteristic. If the values of the expression satisfy the conditions in col (6) of [Table 4](#), the lot shall be declared to have satisfied the requirement for calcium chloride content.

B-5.2 For Composite Samples

The lot shall be deemed to have met the requirements of the characteristics tested on the composite sample if the test results satisfy the corresponding requirements given in [Table 1](#).

B-5.3 The lot shall be considered as conforming to the specification if it satisfies all the criteria given in [B-5.1](#) and [B-5.2](#).

Table 4 Criteria for Conformity for Calcium Chloride Content

(Clauses [B-5.1](#))

Sl No.	Type	Grade	Average of Test Results 1, 2, 3,N	Range	Criteria for Conformity
(1)	(2)	(3)	(4)	(5)	(6)
i)	A	1	\bar{X}_1	R_1	$(\bar{X}_1 - 0.6 R_1)$ shall be greater than or equal to 74.0
ii)	A	2	\bar{X}_2	R_2	$(\bar{X}_2 - 0.6 R_2)$ shall be greater than or equal to 70.0
iii)	A	3	\bar{X}_3	R_3	$(\bar{X}_3 - 0.6 R_3)$ shall be greater than or equal to 70.0
iv)	B	-	\bar{X}_4	R_4	$(\bar{X}_4 - 0.6 R_4)$ shall be greater than or equal to 96.0
v)	C	-	\bar{X}_5	R_5	$(\bar{X}_5 - 0.6 R_5)$ shall be greater than or equal to 90.0

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

<i>Organization</i>	<i>Representative(s)</i>
Central Salt and Marine Chemicals Research Institute, Bhavnagar	DR KANNAN SRINIVASAN (<i>Chairperson</i>)
Alkali Manufacturers Association of India, Delhi	SHRI K. SRINIVASAN SHRI H. S. DAS (<i>Alternate</i>)
Bhabha Atomic Research Centre, Mumbai	DR A. V. R. REDDY DR S. N. ACHARY (<i>Alternate</i>)
Central Drugs Standard Control Organization, New Delhi	DR RAMAN MOHAN SINGH
Consumer Voice, Delhi	SHRI M. A. U. KHAN SHRI K. C. CHAUDHARY (<i>Alternate</i>)
Delhi Jal Board, New Delhi	SHRI ASHUTOSH KAUSHIK
Directorate General of Quality Assurance (DGQA), New Delhi	DR GURBACHAN SINGH SHRI B. S. TOMAR (<i>Alternate</i>)
Geological Survey of India, Kolkata	SHRI PVVR SARMA
Global Adsorbents Pvt Ltd, Kolkata	SHRI SANJAY DHANUKA SHRI RAHUL DHANUKA (<i>Alternate</i>)
Grasim Industries Ltd, Nagda	SHRI ALOK SINGH SHRI PANKAJ GUPTA (<i>Alternate</i>)
Gujarat Alkalies and Chemicals Ltd, Vadodara	SHRI V. K. MAHIDA SHRI SHAILESH PATEL (<i>Alternate</i>)
Hindalco, Mumbai	SHRI NAGESWAR KAPURI SHRI AJITH RAMACHANDRA (<i>Alternate</i>)
Hindustan Lever Ltd, Mumbai	MS VRINDA RAJWADE SHRI SOJAN VARGHESE (<i>Alternate</i>)
Indian Chemical Council (ICC), New Delhi	DR U. SHETKAR DR RAKESH KUMAR (<i>Alternate</i>)
Indian Institute of Chemical Technology, Hyderabad	DR PRAVEEN R. LIKHAR DR RAJENDER REDDY (<i>Alternate</i>)
Industrial Carbon Pvt Ltd, Ankleshwar	SHRI SATYAN ROHIT KUMAR
Ministry of Chemicals and Fertilizers, New Delhi	DR ROHIT MISRA DR O. P. SHARMA (<i>Alternate</i>)
Ministry of Defence (DGQA), Kanpur	SHRI R. N. APARAJIT
National Chemical Laboratory, Pune	DR DARBHA SRINIVAS DR PARESH DHEPE (<i>Alternate</i>)
National Metallurgical Laboratory, Jamshedpur	DR TRILOCHAN MISHRA SHRI DEVBRATA MISHRA (<i>Alternate</i>)

IS 1314 : 2024

National Mineral Development Corporation Ltd, Hyderabad	SHRI RAJAN KUMAR DR PRASHANT SHARMA (<i>Alternate</i>)
National Physical Laboratory, New Delhi	DR NAHAR SINGH DR S. P. SINGH (<i>Alternate</i>)
National Peroxide Ltd, Mumbai	DR JOY ANTHONY
National Test House, Ghaziabad	MS RICHA KUNDRA
Office of the Development Commissioner (MSME), New Delhi	DR KARTHIKEYAN MS ANNA BACKIAM (<i>Alternate</i>)
Shriram Institute for Industrial Research, Delhi	DR LAXMI RAWAT SHRI B. GOVINDAN (<i>Alternate</i>)
Tamilnadu Petroproducts Limited, Chennai	SHRI RAVI MUTHUKRISHNAN
Tata Chemicals Ltd, Mithapur	SHRI NAJMUL HASAN KHAN
The Dharamsi Morarji Chemicals Co Ltd, Mumbai	SHRI MANDAR GAIKWAD
Vaibhav Analytical Services, Ahmedabad	SHRI GAURANG OZA
In Personal Capacity (<i>Hari Nagar Co-Op-Society, Gotri Road, Vadodara - 390007</i>)	SHRI R. S. BAGHEL
In Personal Capacity (<i>514 Veer Apt, Sector 13, Rohini, New Delhi - 110085</i>)	SHRI D. K. JAIN
BIS Directorate General	SHRI A. K. LAL, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (CHEMICAL) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary
SHRI SAGAR SINGH
SCIENTIST 'D'/JOINT DIRECTOR
(CHEMICAL), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 2016* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Head (Publication & Sales), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: CHD 01 (24521).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002

Telephones: 2323 0131, 2323 3375, 2323 9402

Website: www.bis.gov.in

Regional Offices:

	Telephones
Central : 601/A, Konnectus Tower -1, 6 th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	{ 2323 7617
Eastern : 8 th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	{ 2367 0012 2320 9474
Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	{ 265 9930
Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	{ 2254 1442 2254 1216
Western : Manakalya, 5 th Floor/MTNL CETTM, Technology Street, Hiranandani Gardens, Powai Mumbai 400076	{ 25700030 25702715

Branches : AHMEDABAD, BENGALURU, BHOPAL, BHUBANESHWAR, CHANDIGARH, CHENNAI, COIMBATORE, DEHRADUN, DELHI, FARIDABAD, GHAZIABAD, GUWAHATI, HARYANA (CHANDIGARH), HUBLI, HYDERABAD, JAIPUR, JAMMU, JAMSHEDPUR, KOCHI, KOLKATA, LUCKNOW, MADURAI, MUMBAI, NAGPUR, NOIDA, PARWANOO, PATNA, PUNE, RAIPUR, RAJKOT, SURAT, VIJAYAWADA.