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*Draft Indian Standard*  
**MAGNESIUM CHLORIDE — SPECIFICATION**  
*(Third Revision of IS 254)*

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

**Last date of comments: 17<sup>th</sup> December 2022**

## FOREWORD

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

This standard was originally published in 1950 and subsequently revised in 1962 and 1973. In the previous revision, the pharmaceutical grade of the material was dropped since it is covered under Indian Pharmacopoeia and another grade has been introduced to cover the material for analytical purposes. Also Grades 2 and 3 were combined into one and now the Grade 3 covers the material for textile and flooring compositions. The requirements and the methods of test for all the grades have been reviewed completely and the sampling scheme modified.

In this revision, instrumental test methods for the determination of arsenic, calcium, iron, lead, manganese and sulphates have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, amendment no. 1 and Reference clause have been incorporated.

Magnesium chloride is used in the manufacture of magnesium salts, magnesium cements, paper, road dust laying compounds and flooring compositions. It is also used in disinfectants; fire extinguishers; for fireproofing of wood; in refrigerating brines; cooling of drilling tools; in textile industry for sizing, dressing and filling of cotton and woollen fabrics, for thread lubrication, carbonization of wool; as additive in casein glue; and as a reagent in analytical chemistry.

For particle size IS Sieves (*see* to IS 460) are prescribed. Where IS Sieves are not available other equivalent standard sieves as judged by aperture size may be used.

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.

## 1 SCOPE

**1.1** This standard prescribes the requirements and the methods of sampling and test for magnesium chloride.

## 2 REFERENCES

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

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid — Specification ( <i>third revision</i> )
265 : 2021	Hydrochloric acid — Specification ( <i>fifth revision</i> )
266 : 1993	Sulphuric acid — Specification ( <i>third revision</i> )
296 : 1986	Specification for sodium carbonate, anhydrous ( <i>third revision</i> )
323 : 2009	Rectified spirit for industrial use — Specification ( <i>second revision</i> )
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
2088 : 1983	Methods for determination of arsenic ( <i>second revision</i> )
4161 : 1967	Specification for Nessler cylinders
4905 : 2015/	Random sampling and randomization procedures ( <i>first revision</i> )
ISO 24153	
6393 : 1987	Specification for phenylacetamide ( <i>first revision</i> )

## 3 GRADES

**3.1** The material shall be of the following grades:

- Grade 1* — Analytical reagent grade.
- Grade 2* — Pure grade, for use in chemical industry.
- Grade 3* — Technical grade, for use in textile industry and flooring compositions.

## 4 REQUIREMENTS

### 4.1 Description

#### 4.1.1 For Grades 1 and 2

The material of Grades 1 and 2 shall be in the form of colourless, deliquescent crystals. The material shall be free from visible impurities.

#### 4.1.2 For Grade 3

The material of Grade 3 shall be in the form of fused lumps, flakes or crystals. The material shall be free from visible impurities.

**4.2** The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col **6** of the table.

## 5 PACKING AND MARKING

### 5.1 Packing

The material shall be packed in suitable airtight containers as agreed to between the purchaser and the supplier but the material of Grades 1 and 2 should preferably be packed in glass bottles with glass or plastic stoppers. The containers shall be airtight.

**5.2 Marking** — The containers shall be marked with the following information:

- a) Manufacturer's name and recognized trade-mark, if any;
- b) Name and grade of the material;
- c) Mass of the material in the container; and
- d) Batch number and/or lot number, in code or otherwise.

**5.2.1** In the case of analytical reagent grade, the maximum limits for the characteristics given in col **3** of Table 1 shall also be given on the label.

**Table 1 Requirements for Magnesium Chloride**  
(Clause 4.2)

Sl. No.	Characteristic	Requirement			Method of Test (Ref to Clause No. in Annex A)
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Matter insoluble in water, percent by mass, <i>Max</i>	0.003	0.01	—	A-2
ii)	Magnesium chloride (as $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ), percent by mass, <i>Min</i>	98.0	98	95.0	A-3 or A-10
iii)	Sulphate (as $\text{SO}_4$ ), percent by mass	0.002	0.02	1.6	A-4 or A-19
iv)	Calcium (as Ca), percent by mass, <i>Max</i>	0.005	0.2	1.0	A-5 or A-18
v)	Iron (as Fe), percent by mass, <i>Max</i>	0.0002	0.001	—	A-6 or A-18
vi)	Heavy metals (as Pb), ppm, <i>Max</i>	5	5	—	A-7 or A-18
vii)	Arsenic (as As), ppm, <i>Max</i>	1	2	—	A-8 or A-18
viii)	Acidity (as HCl), percent by mass, <i>Max</i>	0.001	To pass test	—	A-9
ix)	Alkalinity (as MgO), ppm, <i>Max</i>	5	—	—	A-10
x)	Alkali chlorides (as NaCl), percent by mass, <i>Max</i>	0.013	1.0	2.0	A-10
xi)	Potassium (as K), percent by mass, <i>Max</i>	0.005	—	—	A-11
xii)	Ammonium salts (as N), percent by mass, <i>Max</i>	0.002	0.02	—	A-12
xiii)	Ethanol insoluble matter	To pass test	To pass test	—	A-13
xiv)	Phosphate (as $\text{PO}_4$ ), ppm, <i>Max</i>	2	—	—	A-14
xv)	Manganese (as Mn), ppm, <i>Max</i>	5	—	—	A-15 or A-18
xvi)	Zinc (as Zn), percent by mass, <i>Max</i>	0.0025	—	—	A-16
xvii)	Barium (as Ba), percent by mass, <i>Max</i>	0.002	—	—	A-17

### 5.2.2 BIS Certification Marking

The product may also be marked with Standard mark.

**5.2.2.1** 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

**6.1** The methods of drawing representative samples of the material and the criteria for conformity to the requirements of this standard shall be as given in Annex B.

### ANNEX A

(Clause 4.2, and Table 1)

## METHODS OF TEST FOR MAGNESIUM CHLORIDE

### A-1 QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

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

### A-2 DETERMINATION OF MATTER INSOLUBLE IN WATER

#### A-2.1 Procedure

Weigh accurately about 25 g of the material and dissolve in about 250 ml of water. Filter through a tared Gooch crucible or sintered glass crucible (G No. 4). Wash the residue thoroughly with water till it is free from all soluble compounds and dry to constant mass at  $110 \pm 2^\circ\text{C}$ .

#### A-2.2 Calculation

$$\text{Matter, insoluble in water, percent by mass} = 100 \frac{M_1}{M_2}$$

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 TOTAL MAGNESIUM

#### A-3.1 Reagents

##### A-3.1.1 *Standard Magnesium Solution*

Dissolve 2.4640 g of magnesium sulphate heptahydrate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in 1 l of water.

##### A-3.1.2 *Standard EDTA Solution*

Dissolve 3.72 g of disodium ethylene-diamine tetra-acetate dihydrate in water and make the volume to 1 l. The solution shall be frequently standardized against standard magnesium solution as in **A-3.2**.

##### A-3.1.3 *Ammonium Hydroxide-Ammonium Chloride Buffer Solution*

Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonia (sp. gr. 0.92) and 250 ml of water. A mixture of 0.931 g of EDTA and 0.6166 g of magnesium sulphate heptahydrate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) is dissolved in about 50 ml of water. The two solutions are mixed and made up to 1 l.

#### **A-3.1.4 Eriochrome Black T Indicator**

Dissolve 0.1 g of the dye in 20 ml of methanol. The solution is prepared fresh every week.

#### **A-3.2 Procedure**

Weigh accurately 1.0 g of the material and dissolve it in water. Dilute in a graduated flask to 1 l. Transfer with a pipette 10 ml of the solution into a 250 ml conical flask, add 25 ml of water, 5 ml of the buffer solution, 5 drops of the indicator and titrate against standard EDTA solution to a pure blue end-point. Correct the titre value for the presence of calcium for grades 2 and 3 (see A-5.2.2.3).

#### **A-3.3 Calculation**

$$\text{Total magnesium (as Mg), percent by mass} = \frac{121.6 NV}{M}$$

where

$N$  = normality of standard EDTA solution,

$V$  = corrected volume in ml of standard EDTA solution used in the titration, and

$M$  = mass in g of the material taken for the test in A-3.2.

### **A-4 TEST FOR SULPHATES**

#### **A-4.1 For Grades 1 and 2**

##### **A-4.1.1 Apparatus**

**A-4.1.1.1 Nessler cylinders** — 50 ml capacity (see IS 4161).

##### **A-4.1.2 Reagents**

**A-4.1.2.1 Dilute hydrochloric Acid** — approximately 4 N.

**A-4.1.2.2 Barium chloride solution** — 10 percent.

##### **A-4.1.2.3 Standard sulphate solution**

Dissolve 0.1814 g of potassium sulphate in water and make up the volume to 1000 ml. One millilitre of the solution contains 0.1 mg of sulphate (as  $\text{SO}_4$ ).

##### **A-4.1.3 Procedure**

Dissolve 5.0 g of the material for Grade 1 and 0.50 g of the material for Grade 2, in water, and transfer to a Nessler cylinder. Add 1 ml of dilute hydrochloric acid, 10 ml of absolute alcohol, dilute to 45 ml with water, and add 5 ml of barium chloride solution. Stir immediately with a glass rod and allow to stand for 5 min. Carry out a control test in another Nessler cylinder using 1 ml of standard sulphate solution. Allow the contents to stand for 10 to 15 min and compare the turbidity in the two cylinders.

**A-4.1.3.1** The material shall be taken as not having exceeded the limit prescribed in Table 1 if the turbidity produced with the material is not greater than that in the control test.

**A-4.2 For Grade 3****A-4.2.1 Reagents**

**A-4.2.1.1** *Dilute hydrochloric acid* — approximately 4 N.

**A-4.2.1.2** *Barium chloride solution* — 10 percent (m/v).

**A-4.2.2 Procedure**

Transfer about 4 g of the material, accurately weighed, into a 500 ml beaker and dissolve in about 250 ml of water. Add 10 ml of dilute hydrochloric acid and boil. Add to the boiling solution a slight excess of hot barium chloride solution and continue boiling for 2 min to obtain a granular precipitate of barium sulphate. Let it stand for 4 h and filter the solution through a tared sintered glass crucible (G No. 4) or a tared Gooch crucible. Wash the precipitate thoroughly till it is free from chlorides, and dry to constant mass at 105° to 110°C.

**A-4.2.2.1** Excess of barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution by addition of barium chloride in slow stream with stirring minimizes mechanical occlusion of barium chloride and gives a coarse precipitate which is less soluble in acids.

**A-4.2.3 Calculation**

$$\text{Sulphates as (SO}_4\text{)} = \frac{41.155M_1}{M_2}$$

where,

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

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

**A-4.3 Alternative Method**

Sulphates may alternatively be determined by instrumental test method as prescribed in **A-19**.

**A-5 TEST FOR CALCIUM****A-5.1 For Grade 1 Material****A-5.1.1 Apparatus**

**A-5.1.1.1** *Nessler cylinders* — 50 ml capacity (see IS 4161).

**A-5.1.2 Reagents**

**A-5.1.2.1** *Dilute hydrochloric acid*

**A-5.1.2.2** *Ammonium hydroxide solution*

**A-5.1.2.3** *Ammonium oxalate solution* — 10 percent (m/v).

**A-5.1.2.4** *Standard calcium solution*

Dissolve 2.5022 g of calcium carbonate in water alongwith sufficient dilute hydrochloric acid and make up the volume to 1000 ml. One millilitre of this solution contains 1 mg of calcium (Ca).

**A-5.1.2.5 Dilute standard calcium solution**

Dilute 100 ml of the solution as in **A-5.1.2.4** to 1000 ml. One millilitre of this solution contains 0.1 mg of calcium (Ca).

**A-5.1.3 Procedure**

Dissolve 4 g of Grade 1 material in water and take it into a Nessler cylinder and neutralize it with hydrochloric acid using litmus paper. Add ammonium hydroxide solution till the solution is alkaline. Add 10 ml of ammonium oxalate solution, boil and cool. Make up the solution to 50 ml with water. Carry out another test in another Nessler cylinder using 2 ml of diluted standard calcium solution for Grade 1 in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture and compare the opalescence produced in the two cylinders after 5 min.

**A-5.1.3.1** The limits prescribed in Table 1 for calcium shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

**A-5.2 For Grades 2 and 3 Material (Patton and Reeder's Indicator Method)****A-5.2.1 Reagents**

**A-5.2.1.1 Potassium hydroxide solution** — Approximately 10 N. The solution shall be freshly prepared.

**A-5.2.1.2 Patton and Reeder's indicator mixture**

Mix 0.1 g of 2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthylazo)-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 bottle.

**A-5.2.1.3 Standard EDTA Solution 0.02 M**

Weigh 7.444 g of disodium ethylene diamine tetraacetate dihydrate (EDTA) and dissolve in 500 ml of water. Make up the volume to 1 l. Mix thoroughly.

**A-5.2.1.4 Potassium cyanide**

**A-5.2.1.5 Hydroxylamine hydrochloride** — See IS 323.

**A-5.2.1.6 Calcein indicator**

Grind thoroughly 0.1 g of calcein and 0.06 g of thymolphthalein with 10 g of potassium chloride.

**A-5.2.1.7 Ammonium chloride-ammonium hydroxide buffer solution**

Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (sp. gr. 0.90) and 250 ml of water. Also dissolve separately a mixture of 0.931 g of disodium ethylene diamine tetra-acetate dihydrate and 0.616 g of magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in about 50 ml of water. Mix the two solutions and dilute to 1 l.

NOTE — Five millilitres of buffer solution added to 50 ml of distilled water should not consume more than a drop of EDTA solution to change to distinct blue with eriochrome black T indicator.

**A-5.2.2 Procedure****A-5.2.2.1 Standardization of EDTA solution**



Transfer 25 ml of standard calcium solution into a conical flask, add 25 ml of water, 10 ml ammonium chloride ammonium hydroxide buffer solution, 5 drops of the eriochrome black T indicator solution and titrate against the standard EDTA solution to a pure blue end-point.

**A-5.2.2.2** Titrate 25 ml of the buffer solution with EDTA solution using eriochrome black T indicator. Subtract the buffer correction for 10 ml (usually it will be 0.1 ml) from the reading obtained in **A-5.2.2.1** and note the final titre value. Calculate the calcium equivalent of 1 ml of EDTA solution (say A).

**A-5.2.2.3** Transfer exactly 100 ml of the solution preserved in **A-3.2** into 250 ml conical flask and 5 ml sodium hydroxide solution and stir well. Add 0.2 g murexide ( or 100 mg calcein mixed indicator ) and titrate against standard EDTA solution till wine red colour of the solution changes to pure blue end-point with murexide (or green to purple with mixed calcein indicator) . Note the volume of the standard EDTA solution used in the titration.

### A-5.2.3 Calculation

$$\text{Calcium (as Ca), percent by mass (on dry basis)} = \frac{AV_1 \times 100}{M}$$

where

A = calcium equivalent in mg of 1 ml of EDTA solution determined in **A-5.2.2.2**,

$V_1$  = volume in ml of standard EDTA solution used in **A-5.2.2.3**, and

M = mass in g of the dried sample taken for the test in **A-3.2**.

### A-5.3.1 Procedure

Take a suitable aliquot of the solution of the sample prepared as prescribed in the material specification and containing not more than 20 mg of calcium in a volume of about 50 ml. Take two portions of 50 ml each in two conical flasks or white porcelain dish of about 100 ml capacity. Add about 5 ml of potassium hydroxide solution to one flask. Add about 0.2 to 0.4 g of Patton and Reeder's indicator mixture and titrate quickly till the colour changes "to violet blue. Note the volume of EDTA solution consumed. Add the same volume of EDTA solution 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 4 g of indicator mixture 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 solution consumed in the final titration should be taken for calculating the result.

### A-5.4 Calculation

$$\text{Calcium (as CaO), percent by mass} = 0.08 \frac{V}{M}$$

where

V = volume, in ml, of 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-5.5 Alternative Method

Calcium may alternatively be determined by instrumental test method as prescribed in **A-18**.

**A-6 TEST FOR IRON****A-6.1 Apparatus**

**A-6.1.1 Nessler Cylinders** — 50 ml capacity (*see* IS 4161).

**A-6.2 Reagents**

**A-6.2.1 Hydrochloric Acid** — sp. gr. 1.16 (*See* IS 265).

**A-6.2.2 Ammonium Persulphate**

**A-6.2.3 Butanolic Potassium Thiocyanate**

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-6.2.4 Dilute Sulphuric Acid** — approximately 5 N.

**A-6.2.5 Standard Iron Solution**

Dissolve 0.702 g of ferrous ammonium sulphate [ $\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$ ] in 100 ml of distilled water and 10 ml of concentrated sulphuric acid, add dilute potassium permanganate solution dropwise (0.2 percent, *m/v*) until a slight pink colour persists after stirring. And then dilute with water to 1000 ml mark. Transfer 100 ml of this solution to 1000 ml volumetric flask and dilute again to 1000 ml mark. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe).

**A-6.2.5.1 Diluted standard iron solution**

Dilute 100 ml of the solution to 1000 ml. One millilitre of the diluted solution contains 0.001 mg of iron (as Fe).

**A-6.2.6 Prepared Sample Solution**

Quickly transfer about 500 g of the material from the sample container to a tared glass-stoppered weighing bottle, stopper and weigh. Remove the weighed sample to a stoppered flask using freshly boiled and cooled water. Rinse the weighing bottle, adding the rinsings to the flask. Swirl the flask to dissolve the sample, cooling it in water if necessary. Dilute the solution in a graduated flask to 1 l. Allow to stand for 3 h. Dilute 100 ml of the clear supernatant solution to 1000 ml. The diluted solution shall be the prepared solution to be used in the tests.

**A-6.3 Procedure**

Transfer 20 ml of the prepared sample solution in a Nessler cylinder and make acidic with hydrochloric acid. Boil for 1 min and cool to room temperature. Add 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate. Shake vigorously and allow the liquids to separate. Carry out a control test using 2 ml of the diluted standard iron solution for Grade 1 and 10 ml of standard solution for Grade 2 and the same quantities of the other reagents in the same total volume of the reaction mixture.

**A-6.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the red colour produced in the test with the material is not greater than that produced in the control tests.

**A-6.4 Alternative Method**

Iron may alternatively be determined by instrumental test method as prescribed in **A-18**.

## **A-7 TEST FOR HEAVY METALS (AS LEAD)**

### **A-7.1 Apparatus**

**A-7.1.1 Nessler Cylinders** — 50 ml capacity (*see* IS 4161).

### **A-7.2 Reagents**

**A-7.2.1 Acetic Acid** — approximately 33 percent.

**A-7.2.2 Dilute Ammonium Hydroxide** — approximately 4 N.

#### **A-7.2.3 Potassium Cyanide Solution**

Dissolve 10 g of potassium cyanide in 90 ml of water, add 2 ml of hydrogen peroxide (20 volume strength), allow to stand for 24 h and make up to 100 ml with water.

**Caution** — Potassium cyanide is highly poisonous and hence handle with care.

**A-7.2.4 Sodium Sulphide Solution** — Dissolve 10 g of sodium sulphide in 100 ml of water.

#### **A-7.2.5 Standard Lead Solution**

Dissolve 0.160 g of lead nitrate in 5 ml of concentrated nitric acid (*See* IS 264) and dilute to 100 ml in a graduated flask. Again dilute 10 ml of the solution to 1000 ml. One millilitre of the solution finally obtained contains 0.01 mg of lead (as Pb).

### **A-7.3 Procedure**

Dissolve 5.000 g of the material in water in a Nessler cylinder and add 5 ml of acetic acid. Make the mixture alkaline with dilute ammonium hydroxide and add 1 ml of potassium cyanide solution. If turbid, filter. Dilute the clear solution obtained to mark, add two drops of sodium sulphide solution and mix well. Carry out a control test in another Nessler cylinder in exactly the same manner using 2.5 ml of standard lead solution for Grades 1 and 2 in place of the material, Compare the colour produced in the two cylinders against a white background.

**A-7.3.1** The material shall be taken to have not exceeded the limits specified in Table 1 if the intensity of colour obtained with the material is not greater than that obtained in the control test.

### **A-7.4 Alternative Method**

Lead may alternatively be determined by instrumental test method as prescribed in **A-18**.

## **A-8 TEST FOR ARSENIC**

### **A-8.1 Gutzeit Method**

Weigh accurately 1.000 g of the material of Grade 1 and 0.5 g of Grade 2 and dissolve in 10 ml of water and carry out the test for arsenic as prescribed in IS 2088 using 0.001 32 mg of arsenic trioxide ( $\text{As}_2\text{O}_3$ ) for preparing the comparison stain.

### **A-8.2 Alternative Method**

Arsenic may alternatively be determined by instrumental test method as prescribed in **A-18**.

**A-9 TEST FOR ACIDITY****A-9.1 For Grade 1 Material****A-9.1.1 Reagents****A-9.1.1.1 Phenolphthalein indicator**

Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit (*See* to IS 323), 95 percent by volume that has been previously neutralized to the indicator.

**A-9.1.1.2 Standard potassium hydroxide solution** — 0.5 N and 0.2 N.

**A-9.1.1.3 Standard hydrochloric acid** — 0.5 N and 0.2 N.

**A-9.1.1.4 Potassium fluoride solution**

Dissolve 100 g of potassium fluoride in 120 ml of hot water, freshly distilled. Add 1 ml of phenolphthalein indicator and neutralize with standard hydrochloric acid (0.5 N) or standard potassium hydroxide solution (0.5 N), as the case may be. (One milliliter of potassium fluoride solution in 10 ml of freshly distilled water shall give a faint pink colour,) Filter the solution, if necessary, and then dilute to 200 ml with freshly distilled water.

**A-9.1.2 Procedure**

Pipette out 40 ml of standard hydrochloric acid (0.2 N) into a 100 ml volumetric flask. Add to the flask about 10 g of the material accurately weighed, and shake well to dissolve. Make up the volume to the mark. Pipette out 25 ml of this solution in a 250 ml conical flask. Add 50 ml of potassium fluoride solution and 0.5 ml of phenolphthalein indicator, and cool the contents to 15°C. Titrate the solution with standard potassium hydroxide solution (0.2 N) until a delicate pink colour persisting for 1 min is obtained.

**A-9.1.2.1** Carry out a blank test using 10 ml of standard hydrochloric acid and other reagents.

**A-9.1.3 Calculation**

$$\text{Acidity (as HCl), percent by mass} = \frac{14.60 (V_1 - V_2) N}{M}$$

where

$V_1$  = volume in ml of standard potassium hydroxide solution used in the test with the material,

$V_2$  = volume in ml of standard potassium hydroxide solution used in the blank,

$N$  = normality of potassium hydroxide solution, and

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

**A-9.2 For Grade 2 Material****A-9.2.1 Reagents**

**A-9.2.1.1 Standard sodium hydroxide solution** — 0.1 N.

**A-9.2.1.2 Bromothymol blue indicator**

Dissolve 0.1 g of the bromothymol blue in 100 ml of ethyl alcohol (50 percent v/v).

**A-9.2.2 Procedure**

Weigh accurately 2.0 g of the material and dissolve in 50 ml of freshly boiled and cooled water. Titrate with standard sodium hydroxide solution using bromothymol blue indicator till the solution turns green (pH 7).

**A-9.2.2.1** The material shall be taken to have satisfied the test if not more than 0.05 ml of standard sodium hydroxide solution is required for the tests.

**A-10 COMPUTATION OF RESULTS FOR MAGNESIUM CHLORIDE, MAGNESIUM OXIDE AND ALKALI CHLORIDES****A-10.1 Determination of Total Chlorine****A-10.1.1 Reagents**

**A-10.1.1** Dilute nitric acid — approximately 4 N.

**A-10.1.1.2** Silver nitrate solution — 5 percent.

**A-10.1.2 Procedure**

Dissolve about 0.5 g of the material, accurately weighed, in 100 ml of distilled water and add 20 ml of dilute nitric acid. Heat the solution to 50°C and add to the hot solution sufficient volume of silver nitrate solution to ensure complete precipitation of chlorides. Heat to boiling to coagulate the precipitated silver chloride. Protect the silver chloride precipitate from light by wrapping black paper around the container. Filter through a tared Gooch crucible or a tared sintered glass crucible (G No. 4), and wash the precipitate with cold distilled water. Dry the crucible and its contents to constant mass at about 130°C.

**A-10.1.3 Calculation**

$$\text{Total chlorine ( as Cl ), percent by mass} = \frac{24.74 A}{M}$$

where

$A$  = mass in g of the precipitate, and

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

**A-10.2 Computation of Results**

**A-10.2.1** Calculate the magnesium equivalent of sulphate by multiplying the percentage of sulphates (as obtained in **A-4**) with 0.2533. Subtract the magnesium equivalent of sulphates so obtained from total magnesium obtained in **A-3**. Let this value be  $x$ .

**A-10.2.2** If the total chlorine obtained in A-10.1 is greater the value of  $2.916x$ , then:

a) Magnesium chloride ( as  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  ), percent by mass =  $8.361x$

b) Alkali chlorides ( as  $\text{NaCl}$  ), percent by mass =  $1.649$  (total chlorine  $2.916x$ )

c) Magnesium oxide ( as  $\text{MgO}$  ) = Nil

**A-10.2.3** If the total chlorine obtained in **A-10.1** is less than the value of  $2.916x$ , then:

- a) Magnesium chloride ( as  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  ), percent by mass =  $2.864x$  total chlorine,
- b) Subtract the magnesium equivalent of chlorine, obtained by multiplying the value for total chlorine with 0.3429 from  $x$  and multiply the residual magnesium with 1.658 to obtain magnesium oxide (as  $\text{MgO}$ ), percent by mass, and
- c) Alkali chlorides (as  $\text{NaCl}$ ) is nil.

## **A-11 TEST FOR POTASSIUM**

**A-11.1 Simple Flame Photometer** — equipped with interference filter.

### **A-11.2 Reagents**

#### **A-11.2.1** *Standard Potassium Solution*

Weigh exactly 1 g of potassium chloride, dissolve in distilled water and dilute to 1 l with distilled water in measuring flask. This solution contains 0.1 g of potassium chloride per 100 ml.

#### **A-11.2.2** *Calibration Graph*

Take 10, 20, 40, 60 and 80 ml of standard potassium solution (*see A-11.2.1*) and dilute to 100 ml with distilled water in different measuring flasks. Each flask now contains 0.01, 0.02, 0.04, 0.06 and 0.08 g of potassium chloride per 100 ml. Use these dilute solutions to obtain a corresponding galvanometer reading as given in the procedure **A-11.3** and plot the concentration against galvanometer reading in a rectangular co-ordination graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.01 to 0.1 percent potassium chloride.

#### **A-11.2.3** *Sample Solution*

Dissolve approximately 10 g of the sample in minimum quantity of distilled water and dilute to 100 ml in a measuring flask with distilled water.

### **A-11.3 Procedure**

**A-11.3.1** Use a flame photometer equipped with atomizer, burner; optical selective device consisting of the reflectors, lenses and diaphragms; and measuring instrument consisting of photocell, amplifier and sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element.

**A-11.3.2** Insert the potassium filter corresponding to wave-length 767 nm light burner fed by illuminating gas (laboratory gas) and adjust the specified air pressure between 0.5 to 0.6 kg/cm<sup>2</sup> and maintain the above air pressure constant such that flame is non-luminous by turning the control knob. First spray distilled water and adjust the pointer to zero in galvanometer scale by zero adjustment knob.

Then spray the potassium chloride standard solution (**A-11.2.1**) and adjust the deflection to maximum (100) by using sensitivity control knob. Again spray water to see pointer comes to zero; then spray standard solution to indicate 100. Repeat till water reads zero and standard solution reads 100 with same adjustment during both the operations. Now reading zero by water and with the same adjustment 100 by standard solution indicate that the instrument has been now ready for measurement.

**A-11.3.3** Without altering the earlier adjustment of the instrument, spray various diluted solutions prepared in **A-11.2.2** and obtain a calibration graph in the range 0.01 to 0.1 percent potassium chloride. After washing with distilled water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of potassium chloride in the solution (say A).

#### **A-11.4 Calculation**

$$\text{Potassium, percent by mass} = \frac{52.35 A}{M}$$

where

A = concentration of potassium chloride in the sample solution, and

M = mass of dried sample taken for the test.

### **A-12 TEST FOR AMMONIUM SALTS**

#### **A-12.1 Apparatus**

**A-12.1.1** *Nessler Cylinders* — 50 ml capacity (IS 4161).

#### **A-12.2 Reagents**

**A-12.2.1** *Sodium Hydroxide Solution* — 10 percent.

**A-12.2.2** *Devarda's Alloy* — containing 45 parts aluminium; 50 parts copper and 5 parts zinc.

**A-12.2.3** *Nessler Solution*

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly while stirring, saturated mercuric chloride solution until a slight permanent precipitate is formed. Add 30 g of potassium hydroxide and, when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant the clear solution and keep it in a dark bottle closed with a well-fitting rubber stopper.

NOTE — The solution should be allowed to stand for several hours and filtered only just before use.

**A-12.2.4** *Standard Nitrogen Solution*

Dissolve 0.3819 g of A.R. Grade ammonium chloride in enough water to make up the volume to 1000 ml. Further dilute 100 ml to 1000 ml. One millilitre of this contains 0.01 mg of nitrogen (as N).

#### **A-12.3 Procedure**

Dissolve 1.000 g of the material for Grade 1 and 0.1 g for Grade 2 in 40 ml of water. Add 1 g of powdered Devarda's alloy and 10 ml of 10 percent sodium hydroxide solution. Allow to stand for 2 h protected from loss or access to ammonia, then distil 40 ml, collecting the distillate in 5 ml of water containing 1 drop of dilute hydrochloric acid (*see* Fig. 1 of IS 6393). Transfer the distillate to a Nessler cylinder, and add 1 ml of 10 percent sodium hydroxide solution and 2 ml of Nessler solution. Carry out control tests in exactly the same manner using 2 ml of standard nitrogen solution for Grade 1 and Grade 2.

**A-12.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control tests.

### **A-13 TEST FOR ETHANOL INSOLUBLE MATTER**

### A-13.1 Apparatus

A-13.1.1 *Nessler Cylinder* — 50 ml capacity (IS 4161).

### A-13.2 Reagents

A-13.2.1 *Rectified Spirit* — See IS 323.

### A-13.3 Procedure

Dissolve 5 g of the material in 30 ml of industrial methylated spirit contained in a Nessler cylinder.

A-13.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if a clear colourless solution is obtained.

## A-14 TEST FOR PHOSPHATES

### A-14.1 Apparatus

A-14.1.1 *Nessler Cylinders* — 50 ml capacity (IS 4161).

### A-14.2 Reagents

A-14.2.1 *Dilute Sulphuric Acid* — approximately 5 N.

A-14.2.2 *Phosphate Reagent No. 1*

Dissolve 5 g of ammonium molybdate in 100 ml of 1 N sulphuric acid.

A-14.2.3 *Phosphate Reagent No. 2*

Dissolve 0.2 g of methyl-*p*-aminophenol sulphate (metol) and 20 g of potassium metabisulphite in 100 ml of water.

A-14.2.4 *Standard Phosphate Solution*

Dissolve 1.43 g of potassium dihydrogen phosphate ( $\text{KH}_2\text{PO}_4$ ) in water and dilute to 1000 ml. Store in a polyethylene bottle. Dilute 1 ml of this solution to 1000 ml in a measuring flask just before use. One millilitre of the diluted solution contains 0.001 mg of phosphate (as  $\text{PO}_4$ ).

### A-14.3 Procedure

Dissolve 2.00 g of the material in 20 ml of water in a platinum dish, neutralize with about 10 ml of dilute sulphuric acid and add 2 ml of the acid in excess and dilute to 50 ml.

A-14.3.1 Transfer 25 ml of the above solution into a Nessler cylinder. Add 2 ml of dilute sulphuric acid, 1 ml of phosphate reagent No. 1, 1 ml of phosphate reagent No. 2 and dilute to 50 ml with water. Carry out a control test in the other Nessler cylinder using 2 ml of standard phosphate solution and the same quantities of other reagents in the same volume of the reaction mixture. Keep both the cylinders in a water-bath at 60°C for 10 min and then compare the colour produced in both the cylinders after 5 min.

A-14.3.2 The limit prescribed for phosphates shall be taken as not having been exceeded if any blue colour developed in the test with the material is not deeper than that produced in the control test.

## A-15 TEST FOR MANGANESE



**A-15.1 Principal** — Manganese is determined calorimetrically by visual comparison.

**A-15.2 Apparatus**

**A-15.2.1 Nessler Cylinder** — 100 ml capacity (*see* IS 4161).

**A-15.3 Reagents**

**A-15.3.1 Dilute Nitric Acid** — 1 : 1 (v/v) .

**A-15.3.2 Sodium Carbonate** — *See* to IS 296.

**A-15.3.3 Dilute Phosphoric Acid** — 1 : 1 (v/v).

**A-15.3.4 Potassium Periodate** — solid.

**A-15.3.5 Standard Manganese Solution**

Dissolve 0.3076 g of manganese sulphate monohydrate ( $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ ) in water, add 1 ml of concentrated sulphuric acid (*see* IS 266) and make up the volume to 1000 ml in a volumetric flask. Dilute 10 ml of this solution to 1000 ml. One millilitre of this solution is equivalent to 0.001 mg of manganese (as Mn).

**A-15.4 Procedure**

Weigh accurately about 1 g of the material and dissolve in about 10 ml of dilute nitric acid. Filter and wash residue with hot water. Ignite the residue in a platinum dish, fuse with a little sodium carbonate, dissolve in dilute nitric acid and add to the main filtrate. To this solution add 5 ml of dilute phosphoric acid and 0.6 to 0.8 g of potassium periodate. Heat the solution to boiling and then boil for 20 min. Cool the solution to room temperature. Transfer the solution completely to a Nessler cylinder, make up the volume to 100 ml mark. Carry out a control test in another Nessler cylinder using 5 ml of standard manganese solution in place of the material.

**A-15.4.1** The material shall be taken to have passed the test if the intensity of colour produced with the material is not greater than that produced in the control test.

**A-15.5 Alternative Method**

Manganese may alternatively be determined by instrumental test method as prescribed in **A-18**.

**A-16 TEST FOR ZINC**

**A-16.1 Apparatus**

**A-16.1.1 Nessler Cylinders** — 50 ml capacity (*see* IS 4161).

**A-16.2 Method A**

**A-16.2.1 Reagents**

**A-16.2.1.1 Dilute sulphuric acid** — approximately 5 N, zinc-free.

**A-16.2.1.2 Ammonium thiocyanate solution**

Dissolve 57 g of ammonium thiocyanate (zinc-free) in 100 ml of water.

**A-16.2.1.3 p-Dimethyl amino styryl  $\beta$ -naphthiazole methyl iodide solution**

Approximately 0.05 percent (*m/v*) in rectified spirit (*see* IS 323) shall be zinc-free.

#### **A-16.2.2 Procedure**

Weigh 2 g of the material and dissolve in 20 ml of water in a Nessler cylinder and neutralize with dilute sulphuric acid (about 10 ml), adding one drop in excess. Then add one drop of ammonium thiocyanate solution and 0.2 ml of *p*-dimethyl amino styryl  $\beta$ -naphthiazole methyliodide solution. Carry out a control test in another Nessler cylinder using 30 ml of water, one drop of dilute sulphuric acid, one drop of ammonium thiocyanate solution and 0.2 ml *p*-dimethyl amino styryl *p*-naphthiazole methyliodide solution. Make up the volume in the two cylinders to 50 ml and compare the colour produced immediately.

**A-16.2.2.1** The limit prescribed for zinc shall be taken as not having been exceeded if the colour produced immediately in the test with the material is not pink when compared with that produced in the control test.

**A-16.3 Method B** — This method shall be used, when the reagents required for Method A are not available.

#### **A-16.3.1 Reagents**

**A-16.3.1.1 Dilute sulphuric acid** — approximately 5 N.

**A-16.3.1.2 Dilute hydrochloric acid** — approximately 5 N.

**A-16.3.1.3 Potassium ferrocyanide solution**

Dissolve about 4.2 g of potassium ferrocyanide in 100 ml of water.

#### **A-16.3.2 Procedure**

Weigh 0.5 g of the material and dilute to 5 ml in a Nessler cylinder and neutralize with dilute sulphuric acid (about 2.5 ml), adding one drop in excess. Dilute with water to 30 ml. Add 1 ml of dilute hydrochloric acid and 1 ml of potassium ferrocyanide solution. Make up the volume to 50 ml. Carry out a control test in the other Nessler cylinder using the same quantities of reagents only in the same total volume of the reaction mixture. Allow both the solutions to stand for 1 h and compare the opalescence produced.

**A-16.3.2.1** The limit prescribed for zinc shall be taken as not having been exceeded, if the opalescence produced in the test with the material is not greater than that produced in the control test.

### **A-17 TEST FOR BARIUM**

#### **A-17.1 Apparatus**

**A-17.1.1 Nessler Cylinders** — 100 ml capacity (*see* IS 4161).

#### **A-17.2 Reagents**

**A-17.2.1 Dilute Hydrochloric Acid** — 1 N.

**A-17.2.2 Barium Chloride Solution** — approximately 10 percent (*m/v*).

**A-17.2.3 Dilute Sulphuric Acid** — 5 N.

**A-17.2.4 Rectified Spirit** — *see* IS 323.

**A-17.2.5 Standard Sulphate Solution**

Dissolve 0.148 g of ignited sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) in water and dilute to 1000 ml. One millilitre of this solution contains 0.1 mg of sulphate ( $\text{SO}_4$ ).

**A-17.3 Procedure**

Dissolve 3.5 g of the material in 40 ml of water and add 10 ml of rectified spirit, mix, add 0.5 ml of dilute sulphuric acid, mix immediately and set aside for 1 h. To another Nessler cylinder add 0.5 ml of standard sulphate solution, 37 ml of water and 10 ml of rectified spirit and 1 ml of dilute hydrochloric acid, mix, and add 1 ml of barium chloride solution, mix immediately and allow to stand for 1 h.

**A-17.3.1** The limits 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 with the standard solution.

**A-18 DETERMINATION OF ARSENIC, CALCIUM, LEAD, IRON AND MANGANESE BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD****A-18.1. 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-18.2 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**

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing ( $\mu\text{g}$ )	Axial viewing ( $\mu\text{g}$ )	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Pb	220.353	14	5	Al, Co, Fe, Ti Cr, Fe
		283.305	(70)		
		217.00		(20)	
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	-	-	V, Mo, Zr
iv)	Mn	257.610	1	0.4	Cr, Fe, Mo, W
		293.305	(20)	8	Al, Cr, Fe, Ti
v)	As	188.979	18	14	Al, Cr, Fe, Ti
		193.696	5	14	Al, Co, Fe, W, V
		197.197	(100)	31	Al, Co, Fe, Pb, Ti

**A-18.3 Reagents and Solutions****A-18.3.1 Nitric acid (65 percent) Suprapure****A-18.3.2 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 1000  $\mu\text{g}/\text{ml}$  of Lead, Iron, calcium, manganese and arsenic in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

**A-18.3.3 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, 3.0 and

5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml solution of respective elements under reference.

**A- 18.3.4 Sample preparation**

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

NOTE: Sample should be clear before injecting to the instrument

**A-18.3.5 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-18.4 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.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

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-18.5 Procedure****A-18.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-18.3.5). 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-18.5.2** 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  $\pm 5$  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 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 µg/ml of the lead, Iron, calcium, manganese and arsenic 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-18.6 Calculation**

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

- i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.
- ii) 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-19 ION CHROMATOGRAPHY FOR SULPHATES

### A-19.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of 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, 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-19.2 Equipment

**A-19.2.1** *Anion guard column* — a protector of the separator column.

**A-19.2.2** *Anion Separator column* — suitable for selective separation of ions under analysis.

**A-19.2.3** *Anion Suppressor device* — Anion micromembrane suppressor is used to analyse the data  
Detector: Conductivity detector.

**A-19.2.4** *Software* — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

Sample loop of 100 µl, 200 µl, 500 µl or 1000 µl be used to determine ionic concentration as per instrument manual and practice.

### A-19.3 Reagents and Standards

**A-19.3.1** *Glass or polyethylene sample bottles.*

**A-19.3.2** *Distilled water or deionized water free from the anions of interest.*

**A-19.3.3** *Eluent* — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used.

For preparation of these solution, 0.2856 g of sodium bicarbonate and 0.3816 g of sodium carbonate is dissolved in 2 l of water.

**A-19.3.4 Micromembrane suppressor solution (0.025 N of sulphuric acid)** — Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water

#### **A-19.4 Standard solutions**

**A-19.4.2 Sulphate** — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

#### **A-19.5 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 0.1-1.0 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-19.6 Procedure**

Dissolve between 1 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 (0.1-1.0 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 and deviation of retention force shall not exceed  $\pm 10$  percent of RT of calibration. 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 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-19.7 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 6.1)  
**METHOD OF SAMPLING OF MAGNESIUM 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 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 drawn for sampling shall be mixed thoroughly by suitable means.

**B-1.5** The sample shall be placed in suitable, clean, dry and airtight glass containers.

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

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

All the packages in a single consignment of magnesium chloride 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.

**B-2.1.1** The number of packages ( $n$ ) to be drawn from the lot shall depend upon the size of the lot ( $N$ ) and shall be in accordance with Table 3.

**Table 3 Number of Packages to be selected for Sampling**

<b>Lot Size</b> ( $N$ )	<b>No. of Packages to be Drawn</b> ( $n$ )
4 to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

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

**B-2.1.2** These packages shall be drawn at random from the lot and in order to ensure the randomness of sampling, random number table (*see* IS 4905) may be used.



**B-2.1.3** Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of this specification.

### **B-3 PREPARATION OF TEST SAMPLES**

**B-3.1** From each of the packages selected according to **B-2.1.2**, 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 taken and mixed thoroughly to form a composite sample of about 600 g. The composite samples shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

**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 container samples shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

**B-3.4** All the individual samples and the composite sample shall be transferred to separate sample 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. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of dispute between the two.

### **B-4 NUMBER OF TESTS**

**B-4.1** Test for magnesium chloride content shall be conducted on each of the individual samples.

**B-4.2** Test for the determination of all other characteristics given under **4** shall be performed on the composite sample (*see B-3.2*).

### **B-5 CRITERIA FOR CONFORMITY**

#### **B-5.1 For Individual Samples**

From the test results for magnesium chloride the average ( $\bar{X}$ ) and the range ( $R$ ) or ( $R$ ) shall be computed.

NOTE — Range is defined as the difference between the maximum and minimum of the individual test result & when there are 10 values, they shall be formed into two groups of five each in the same order in which the individual samples are numbered. For each group range shall be calculated and  $R$  is the average of these two  $R$ 's.

**B-5.1.1** The lot shall be considered satisfactory in test of magnesium chloride content if  $(\bar{X} - 0.6 R)$  or  $(\bar{X} - 0.6 R)$  is greater than or equal to 98.0 for Grade 1, 98 for Grade 2 and 95.0 for Grade 3.

#### **B-5.2 For Composite Samples**

The lot shall be considered to have passed in respect of the characteristics tested on the composite test sample, if the test results satisfy the corresponding requirements given under **4**.

**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**.