

भारतीय मानक मसौदा  
रासायनिक उद्योगों के लिए सामान्य नमक – विशिष्टि  
(आईएस 797 का चौथा पुनरीक्षण)

*Draft Indian Standard*  
**COMMON SALT FOR CHEMICAL INDUSTRIES — SPECIFICATION**  
(*Fourth Revision of IS 797*)

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ICS 71.060

Inorganic Chemicals Sectional Committee, CHD 01

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

## FOREWORD

This Indian Standard (Fourth 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 1955 and subsequently revised in 1967. The original version included only one grade of the material. In the first revision another grade of the material required for the manufacture of hydrosulphite and optical brightners was incorporated. The standard was again revised in 1976; in the second revision the requirements of magnesium and sulphate contents was modified. In grade 2 of the material, optional requirements for grit content and trace metals like vanadium, chromium, titanium, molybdenum, copper, nickel and aluminium were incorporated for use of the material in the explosive and pyrotechnic industry and electrolytic purposes respectively. The methods of test for the determination of the trace metals were also incorporated. The sampling scheme was also modified accordingly.

In third revision, additional requirements for the materials of Grade 2, for use in the explosives and pyrotechnic industry for sulphate, iron and alkalinity were incorporated. The method of test for the determination of alkalinity was also incorporated.

In this revision, instrumental test methods for the determination of sulphates, iron, magnesium and calcium have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, Reference clause and amendment have been incorporated in this revision.

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 common salt for chemical industries.

## 2 REFERENCES

**2.1** The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian 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>
250 : 2022	Potassium bichromate technical and analytical reagent — Specification ( <i>second revision</i> )
253 : 2014	Specification for common salt ( <i>fourth revision</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> )
4161 : 1967	Specification for nessler cylinders
4905 : 2015/	Random sampling and randomization procedures ( <i>first revision</i> )
ISO 24153: 2009	
3025(Part 2) :2019/	Methods of sampling and test (Physical And Chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (Icp - Oes) ( <i>first revision</i> )
ISO 11885	
5296 : 1995	Chloroform pure and technical — Specification ( <i>second revision</i> )

## 3 GRADES

**3.1** There shall be two grades of the material, namely:

*Grade 1* — suitable for the manufacture of hydro sulphite and optical brightners or where a pure grade of the material is required.

*Grade 2* — suitable for chemical industries, explosive and pyrotechnic industry and for the manufacture of caustic soda by electrolytic process.

## 4 REQUIREMENTS

### 4.1 Description

The material shall be in the form of large or small colourless crystals, or a white granular powder. The material shall be free from visible contamination with clay, grit and other extraneous adulterants and impurities.

### 4.2 Moisture Content

#### 4.2.1 For Grade 1 Material

It shall contain not more than 1.0 percent moisture, on as received basis, when tested in accordance with the method prescribed in **A-3**.

#### 4.2.2 For Grade 2 Material

It shall contain not more than 4.0 percent moisture, on as received basis, when tested in accordance with the method prescribed in **A-3**.

**4.3** The material shall also comply with the requirements specified in Table 1 when tested according to the methods prescribed in Annex A. Reference to relevant clauses of Annex A is given in col 5 of the table.

**Table 1 Requirements for Common Salt for Chemical Industries**

Sl. No.	Characteristic	Requirement for		Method of Test (Ref to Cl No. In Annex A)
		Grade 1 ( On Dry Basis )	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Sodium chloride ( as NaCl ), percent by mass, Min	99.5	98.5	A-4
ii)	Matter insoluble in water, percent by mass, Max	0.05	0.2	A-5
iii)	Calcium salts (as Ca), percent by mass, Max	0.03	0.2	A-6 or A-15
iv)	Magnesium salts (as Mg), percent by mass, Max	0.01	0.1	A-6 or A-15
v)	Sulphates (as SO <sub>4</sub> ), percent by mass, Max	0.2	0.6	A-7 or A-14
vi)	Iron compound (as Fe), parts per million, Max	10	20	A-8 or A-15

#### 4.3.1 Optional Requirements

**4.3.1.1** For use in the explosives and pyrotechnic industry, the material of Grade 2 shall have grit content of 20 ppm maximum when tested in accordance with the method prescribed in **A-12**.

For use in the explosives and pyrotechnic industry, the material of Grade 2 shall also have the following additional requirements when tested in accordance with the methods prescribed in **A-7**, **A-8** and **A-13**.

- |    |  |                         |
|----|--|-------------------------|
| a) | Sulphate   | 0.5 percent, <i>Max</i> |
| b) | Iron   | 10 ppm, <i>Max</i> and  |
| c) | Alkalinity (as Na <sub>2</sub> CO <sub>3</sub> ) | 0.5 percent, <i>Max</i> |

**4.3.1.2** For the manufacture of caustic soda by the electrolytic process, the material of Grade 2 shall have the following additional requirements when tested in accordance with the methods prescribed in **A-9**, **A-10** and **A-11** of Annex A:

- |                                     | <i>Parts per Million, Max</i> |
|-------------------------------------|-------------------------------|
| a) Vanadium + chromium + molybdenum | 0.01                          |
| b) Aluminium (as Al)                | 0.10                          |

- c) Other trace metals like titanium, copper and nickel 0.01

## 5 PACKING AND MARKING

### 5.1 Packing

The grade 1 material shall be supplied in polyethylene lined jute bags or with a liner; polyethylene lined HDPE bag or with a liner or any other suitable packing as agreed to between the purchaser and the supplier. Grade 2 material shall be packed in jute bags or any other packing as agreed to between the purchaser and the supplier or loose in truck loads country craft or steamers. The packages shall be securely closed.

### 5.2 Marking

The packages shall be marked legibly and indelibly with the following information:

- a) Name and grade of material;
- b) Mass of the material;
- c) Lot or batch number in code or otherwise; and
- d) Name of manufacturer and recognized trade-mark; if any.

#### 5.2.1 BIS Certification Marking

The product may also be marked with the Standard Mark.

**5.2.1.1** 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 thereunder, and the products may be marked with the standard mark.

## 6 SAMPLING

**6.1** The method of preparation of test samples and determination of their criteria for conformity shall be as given in Annex B.

## ANNEX A

(Clauses 4.2.1, 4.2.2, 4.3, 4.3.1.1, 4.3.1.2 and Table 1)

### METHOD OF TEST FOR COMMON SALT FOR CHEMICAL INDUSTRIES

#### A-1 GENERAL

##### A-1.1 Quality of Reagents

Unless specified otherwise, pure chemical 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 PREPARATION OF SAMPLE

**A-2.1** Crush 500 g of the sample to pass through a 1 mm IS sieve, dry in an air oven at about 150 °C for at least 4 h. Cool in a desiccator and transfer to a clean glass stoppered weighing bottle kept in a desiccator for subsequent tests.

**A-2.2** Accurately weigh about 20 g of the dried sample, dissolve it in 200 ml of water in a beaker. Heat to boiling, filter and wash the paper free of chloride. Cool and make up the solution to 1 l. Use this solution for subsequent tests.

**A-2.3** For trace metal analyses, dissolve completely 3.200 g of the salt samples in water, acidify with 100 ml hydrochloric acid and stir well to dissolve all the soluble matter. Filter and make up to 10 l. Use this solution for all trace metal analyses.

**A-2.4** Crush 500 g of the sample to pass through a 1 mm IS Sieve and transfer to a clean glass stoppered weighing bottle kept in a desiccator for subsequent tests.

**A-2.5** Accurately weigh about 20 g of the ground sample and dissolve it in 200 ml of water in a beaker. Heat to boiling, filter and wash the paper free of chloride. Cool and make up the solution to 1 l. Use this solution for subsequent tests.

### **A-3 DETERMINATION OF MOISTURE CONTENT**

#### **A-3.1 Procedure**

Grind rapidly the material, as received (say 100 g), in an agate mortar approximately to a size of 2.8 mm IS Sieve, but do not actually sieve. The material which shall be in the form of powder shall be kept in an air-tight container. Weigh accurately about 20 g of the material in the weighing bottle of about 30 ml capacity, preferably wide mouth squat type, previously dried and weighed. Dry in an oven at 140 to 150°C for at least 4 h. Cool in a desiccator and weigh. Repeat drying, cooling and weighing until constant mass is obtained.

#### **A-3.2 Calculation**

$$\text{Moisture, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where,

$M_1$  = mass in g of the material taken for the test, and

$M_2$  = mass in g of the material after drying.

### **A-4 DETERMINATION OF TOTAL CHLORIDES**

#### **A-4.1 Reagents**

**A-4.1.1** *Potassium Chromate Indicator Solution* — 5 percent.

**A-4.1.2** *Standard Silver Nitrate Solution* — 0.1 N.

#### **A-4.2 Procedure**

Transfer 10 ml of the solution reserved in **A-2.2** into a conical flask and add 1 ml of potassium chromate indicator solution. Titrate against standard silver nitrate solution till the reddish brown tinge persists after brisk shaking. Carry out a blank determination.

### A-4.3 Calculation

$$\text{Total chlorides ( as Cl ), percent by mass} = \frac{3.546 \times VN}{M}$$

where,

$V$  = volume in ml of standard silver nitrate solution used in the titration with the material corrected to blank,

$N$  = normality of standard silver nitrate solution, and

$M$  = mass in g of the dried sample in 10 ml of the solution taken for the test.

**A-4.3.1** Residual chloride is the chloride obtained by deducting the chloride attributed to the other chlorides, such as magnesium chloride and calcium chloride as described in **A-5** of IS 253 from the total chlorides determined in **A-4.3**.

$$\text{Sodium chloride (as NaCl),} = \text{Residual chloride (as Cl)} \times 1.648 \text{ percent by mass}$$

## A-5 DETERMINATION OF MATTER INSOLUBLE IN WATER

### A-5.1 Procedure

Accurately weigh about 20 g of the dried sample (*see A-2.1*), dissolve it in 200 ml of water in a beaker, heat to boiling and cool. Filter the solution through a weighed Gooch or sintered glass crucible (G No. 4) and wash the residue till it is free from chlorides. Dry the crucible along with the insoluble residue to constant mass.

### A-5.2 Calculation

$$\text{Matter insoluble in water} = 100 \times \frac{M_1}{M_2}$$

where,

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

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

## A-6 DETERMINATION OF CALCIUM AND MAGNESIUM

### A-6.1 Reagents

#### A-6.1.1 *Standard Calcium Solution*

Weigh 1.000 g of calcium carbonate, dried at 120°C, and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 l in a graduated flask. One millilitre of the solution is equivalent to 0.400 3 mg of calcium (as Ca).

#### A-6.1.2 *Standard EDTA Solution*

Dissolve 3.72 g of disodium ethylene diamine tetra acetate dihydrate in water and dilute in a graduated flask to 1 l. The solution shall be standardized frequently against standard calcium solution following the procedure given in **A-6.2**.

#### A-6.1.3 *Eriochrome Black T Indicator Solution*

Dissolve 0.1 g of the dye in 20 ml of rectified spirit conforming to IS 323. This solution shall be prepared fresh every week.

**A-6.1.4 Dilute Sodium Hydroxide Solution** — approximately 10 percent.

**A-6.1.5 Murexide Indicator Solution**

Grind 0.2 g of murexide with 10 g of sodium chloride until the mixture is homogenous. 0.2 g of this mixture is suitable for 100 ml of the sample solution.

**A-6.1.6 Calcein Indicator**

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

**A-6.1.7 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-6.2 Procedure**

**A-6.2.1 Standardization of EDTA Solution**

Transfer 25 ml of standard calcium solution into a conical flask, add 25 ml of water, 10 ml of 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-6.2.2** 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-6.2.1** and note the final titre value. Calculate the calcium equivalent of 1 ml of EDTA solution (say A).

**A-6.2.3** Transfer exactly 100 ml of the solution preserved in **A-2.2** into a 250 ml conical flask, add 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of eriochrome black T indicator solution and titrate against standard EDTA solution till wine red colour of the solution changes to pure blue end point. Note the volume of EDTA used in the titration.

**A-6.2.4** Transfer exactly 100 ml of the solution preserved in **A-2.2** into 250 ml conical flask add 5 mg sodium hydroxide solution and stir well. Add 0.2 g murexide ( or 100 ml calcein 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-6.3 Calculation**

$$\text{Calcium (as Ca), percent by mass} = \frac{AV_2}{M}$$

$$\text{Magnesium (as Mg), percent by mass} = 0.6068 \frac{A(V_1 - V_2)}{M}$$

where,

A = calcium equivalent in mg of 1 ml of EDTA solution determined in **A-6.2.2**,

$V_2$  = volume in ml of standard EDTA solution used in **A-6.2.4** (for Ca only),

$M$  = mass in g of the dried sample taken for the test in **A-2.2**, and

$V_1$  = volume in ml of standard EDTA solution used in **A-6.2.3**.

### **A-6.3 Alternative Method**

Calcium and magnesium may alternatively be determined by instrumental test method as prescribed at **A-15**.

## **A-7 DETERMINATION OF SULPHATE**

### **A-7.1 Gravimetric Method**

#### **A-7.1.1 Reagents**

**A-7.1.1.1 Dilute hydrochloric acid** — approximately 4 N.

**A-7.1.1.2 Barium chloride solution** — approximately 10 percent,

#### **A-7.1.2 Procedure**

Dissolve about 10 g of dried sample preserved in **A-2.1** in about 400 ml of water, filter and wash the residue free from soluble salts. Collect the filtrate and washings. Add one drop of methyl orange and 10 ml of dilute hydrochloric acid or more till it is pink and then boil. Add to the boiling solution, 10 to 12 ml of barium chloride solution drop by drop so that the addition is in slight excess and continue boiling for 4 min to obtain a granular precipitate. Allow to stand for 4 h and filter through a weighed sintered glass crucible (G No. 4) or Gooch crucible. Wash the precipitate till free from chloride and dry to constant mass at 105 to 110°C. Alternatively, filter through Whatman filter paper No. 42 and wash till it is free from chloride. Ignite and determine as barium sulphate.

#### **A-7.1.3 Calculation**

$$\text{Sulphate (as SO}_4\text{), percent by mass} = 41.13 \times \frac{M_1}{M_2}$$

where,

$M_1$  = mass in g of barium sulphate, and

$M_2$  = mass in g of dried sample taken for test.

### **A-7.2 Volumetric Method**

#### **A-7.2.1 Reagents**

**A-7.2.1.1 Standard (barium chloride+ Magnesium Chloride) Solution** 0.05 N

Dissolve 4.276 g of barium chloride dihydrate ( $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ) and 3.050 g Magnesium Chloride hexahydrate ( $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ) in water and makeup to 1 l.

**A-7.2.1.2 Dilute hydrochloric acid** — approximately 1 N.

#### **A-7.2.1.3 Standard EDTA solution**

Weigh 3.72 g of disodium ethylene diamine tetra acetate dihydrate in water and dilute in a graduated flask to 1 l. The solution shall be standardized frequently against standard barium chloride solution (**A-7.2.1.1**)

and follow the procedure given in **A-6.2**. One millilitre of standard EDTA solution is equal to 0.001374 g of barium (Ba) or 0.00096 g of sulphate (SO<sub>4</sub>).

**A-7.2.1.4** *Erochrome black T indicator solution* — same as in **A-6.1.3**.

**A-7.2.1.5** *Ammonium chloride-ammonium hydroxide buffer solution* — same as in **A-6.1.7**.

#### **A-7.2.2 Procedure**

**A-7.2.2.1** Pipette out 20 ml of standard (Barium Chloride + Magnesium Chloride) solution into a conical flask, add two drops of hydrochloric acid and 10 ml of ammonium chloride-ammonium hydroxide buffer solution. Dilute it with water to about 50 ml, add five drops of eriochrome black T indicator solution and titrate against standard EDTA solution to the pure blue endpoint. Note the titre value (say *A*)

**A-7.2.2.2** Pipette out 100 ml of the solution preserved in **A-2.2/2.4** and add two drops of hydrochloric acid and heat to gentle boiling. To the hot solution, add 20 ml of standard (Barium Chloride + Magnesium Chloride) solution. Boil gently for about 5 min and then cool to room temperature. The solution will be neutral or slightly acidic when tested with a litmus paper. Add 10 ml of ammonium chloride-ammonium hydroxide buffer solution and five drops of eriochrome black T indicator solution. Titrate against standard EDTA solution to the pure blue endpoint. Note the titre value (say *B*).

#### **A-7.2.3 Calculation**

$$\text{Sulphate (as SO}_4\text{), percent} = \text{by mass (on dry basis)} = 0.96 \frac{A + V_1 - B}{M}$$

where,

*A* = volume in ml of standard EDTA solution used in **A-7.2.2.1**,

*V*<sub>1</sub> = volume in ml of standard EDTA solution used in **A-6.2.3** (total Ca + Mg),

*B* = volume in ml of standard EDTA solution used in titration in **A-7.2.2.2**, and

*M* = mass in g of the dried sample taken for the test in **A-2.2**.

### **A-7.3 Alternative Method**

Sulphates may alternatively be determined by instrumental test method as prescribed at **A-14**.

## **A-8 TEST FOR IRON**

### **A-8.1 Apparatus**

**A-8.1.1** *Nessler Cylinders* — 50 ml capacity (see IS 4161)

### **A-8.2 Reagents**

**A-8.2.1** *Dilute Hydrochloric Acid* — approximately 5 N, free from iron.

**A-8.2.2** *Concentrated Nitric Acid* — see IS 264.

**A-8.2.3** *Potassium Thiocyanate Solution* — 5 percent (*m/v*).

**A-8.2.4** *Dilute Sulphuric Acid* — approximately 5 N.

Dissolve 2.81 g of ferrous ammonium, sulphate [FeSO<sub>4</sub>(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O] in 10 ml of dilute sulphuric acid and dilute with water to 1000 ml. Transfer 10 ml of the solution to 1000 ml volumetric flask and dilute again up to the mark. One millilitre of this solution is equivalent to 0.004 mg of iron (as Fe).

### A-8.3 Procedure

Weigh accurately about 2 g of the material and dissolve it in about 20 ml of water. Add about 5 ml of hydrochloric acid in order to make the solution acidic and 2 ml of nitric acid, and boil to oxidize the iron salts. Cool and transfer to a Nessler cylinder, add 5 ml of potassium thiocyanate solution, dilute to the mark with water and stir well. Into a second Nessler cylinder, add 5 ml of hydrochloric acid, 2 ml of nitric acid, 5 ml standard iron solution for Grade 1 and 10 ml of standard iron solution for Grade 2 material, and 5 ml potassium thiocyanate solution. Dilute to the mark and stir well. Compare the colour of the solution in the two cylinders.

**A-8.3.1** The limit prescribed in Table 1 shall be taken to have not been exceeded if the intensity of colour produced with the sample is not greater than that produced by the standard iron solution.

### A-8.4 Alternative Method

Iron may alternatively be determined by instrumental test method as prescribed at **A-15**.

## A-9 TEST FOR VANADIUM, CHROMIUM AND MOLYBDENUM

**A-9.1** Each of these trace metals is estimated separately and the results added.

### A-9.2 Estimation of Vanadium

#### A-9.2.1 Principle

The vanadium is extracted from a large quantity of sample with 8-hydroxyquinoline, and subsequently determined colourimetrically as the phosphotungstate.

#### A-9.2.2 Reagents

**A-9.2.2.1** *8-hydroxyquinoline reagent* — Dissolve 4 g of the reagent in 100 ml of 10 percent acetic acid.

**A-9.2.2.2** *Sodium acetate* — 5 percent.

**A-9.2.2.3** *Chloroform*

**A-9.2.2.4** *Anhydrous sodium sulphate*

**A-9.2.2.5** *Sodium carbonate, anhydrous* — see IS 296.

**A-9.2.2.6** *Phosphoric acid* — 1 : 2.

**A-9.2.2.7** *Sodium tungstate* — 0.5 N. Dissolve 16.5 g sodium tungstate in 100 ml of water.

#### A-9.2.3 Procedure

Take 4 l of the sample solution (*see A-2.3*) Adjust the pH to 4.5-5.5 by the addition of 50 percent sodium hydroxide solution or concentrated sulphuric acid. Add 80 ml sodium acetate solution and 16 ml 8-hydroxyquinoline reagent. Mix thoroughly, check the pH and maintain it again to 4.5-5.5.

Take 1 l of the above solution in a separating funnel and extract the 8-hydroxyquinoline complex using 20 ml portions of chloroform. Combine the extracts and remove traces of water by adding anhydrous sodium sulphate. Transfer the chloroform extract to a platinum dish and evaporate chloroform at very low heat keeping the dish inside a well ventilated fume chamber. The solution has a tendency to creep up the sides of the dish and hence, if necessary, carefully heat the sides of the dish with a burner.

When dry, place the dish in a muffle furnace at about 800 to 900°C and burn off all the organic matter. Add 5 g anhydrous sodium carbonate and fuse carefully at a temperature of 900 to 1000°C, occasionally swirling the dish to cover the sides also. After about 10 to 15 min, cool the dish, fill up two-third with water and warm gently to dissolve the melt. Transfer to a small beaker and filter through a Whatman filter paper No. 42. Neutralize the filtrate with concentrated sulphuric acid to pH 7. For blank, take 5 g of sodium carbonate in a beaker, dissolve in a small quantity of water and neutralize with concentrated sulphuric acid to pH 7. Add to both beakers enough sulphuric acid to make approximately 0.5 N solution and further add 5 ml phosphoric acid and 2.5 ml sodium tungstate solution. Keep the beakers on a hot-plate and boil gently for 2 min. Cool and make up to 50 ml with water. A pale yellow colour of the phosphotungstate would develop in the sample. Find out the percentage transmittance of the solution, taking the blank for 100 percent transmittance, in the photoelectric colorimeter using a cell of 5 cm light path and a blue 400 nm filter. Find out the corresponding optical density and from the standard graph (see A-9.2.4), calculate the equivalent vanadium content.

#### A-9.2.4 Preparation of Standard Graph

Ignite pure vanadium pentoxide at 500 °C for 1 h and cool it in a desiccator. Dissolve 1.785 0 g of this in a slight excess of sodium hydroxide solution. Neutralize with sulphuric acid and add a few drops in excess and finally dilute to 1000 ml. One millilitre of this solution is equivalent to 1.0 mg of vanadium. Further, dilute 10 ml of this solution to 1000 ml. One millilitre of the diluted solution contains 10 micrograms of vanadium.

Take varying volumes of the diluted solution in 100-ml beakers so as to obtain 5, 10, 25, 50, 75 and 100 micrograms of vanadium. Dilute to about 40 ml and make acidic with concentrated sulphuric acid to make approximately 0.5 N. Add 5 ml phosphoric acid and 2.5 ml sodium tungstate solution to each. Boil for 2 min on a hot-plate, cool and dilute to 80 ml. Also take a blank using 40 ml water. Find out the percentage transmittance on a photoelectric colorimeter using blue 400 nm filter and calculate the corresponding optical densities. Plot the vanadium concentration against the corresponding optical density.

#### A-9.2.5 Calculation

$$\text{Vanadium content, parts per million, in the sample solution} = \frac{\alpha}{4000}$$

$$\text{Therefore, vanadium content } (X_1), \text{ parts per million, in the salt} = 3.125 \times \frac{\alpha}{4000}$$

Where,

a = mass in micrograms of vanadium contained in 4 l of the sample solution.

### A-9.3 Estimation of Chromium

#### A-9.3.1 Principle

Chromium hexavalent is reduced to chromium trivalent by boiling with potassium iodide and sodium sulphite. The precipitate is then co-precipitated with aluminium hydroxide. The aluminium hydroxide is

dissolved in 1 : 1 sulphuric acid, and chromium is oxidized to hexavalent form by bromine water. Chromium is determined colorimetrically by reacting with diphenyl carbazide reagent, which gives a purple colour with chromium.

### **A-9.3.2 Reagents**

**A-9.3.2.1 Sulphuric acid** — 1 : 1 (v/v) and 1 : 9 (v/v).

**A-9.3.2.2 Phosphoric acid** — 1 : 5 (v/v).

### **A-9.3.2.3 Aluminium solution**

Dissolve 1 g of aluminium metal in hydrochloric acid and dilute to 1 l.

### **A-9.3.2.4 Diphenyl carbazide reagent**

Dissolve 0.25 g of diphenyl carbazide and 0.4 g of phthalic anhydride in 100 ml ethanol. The solution is stable for one month.

**A-9.3.2.5 Bromine water.**

**A-9.3.2.6 Potassium iodide** — 16 percent solution.

**A-9.3.2.7 Sodium hydroxide solution** — 30 percent.

**A-9.3.2.8 Neutral sodium sulphite.**

**A-9.3.2.9 Potassium dichromate** — See IS 250.

### **A-9.3.3 Procedure**

Take 2 l of the sample solution (*see A-2.3*) in a 4 l beaker, add 40 ml of potassium iodide solution, 40 ml sulphuric acid and 200 ml water. Boil for about 10 min. Add sodium sulphite, in small portions, to decolorize the solution. In case, the solution turns brown on further boiling, continue adding sodium sulphite till the decolorization is stable and brown colour of iodine does not reappear on boiling. Add 30 ml of aluminium solution and stir well. Further, add sodium hydroxide solution dropwise, stir the solution well and adjust the pH to 6.0-6.5. Allow to settle to complete precipitation. Siphon out the clear solution and collect the aluminium precipitate in a small beaker. Filter through a sintered glass crucible and wash once with hot water. Dissolve the precipitate by pouring 60 ml boiling sulphuric acid (1 : 9) into the crucible, apply suction after 2 to 3 min and again wash with hot water.

Collect the filtrate separately in a 250 ml beaker. Add 4 ml bromine water and stir well. Decolorize the yellow colour with a few drops of sodium hydroxide solution and add 1 ml in excess. Boil for 5 min and add 1 : 1 sulphuric acid until the brownish colour appears. Add 2 ml of the acid in excess and boil again till the brownish colour disappears. Cool and add 1 ml phosphoric acid. Make up to 100 ml and divide into two parts. Keep one as a blank and in the other add 1 ml diphenyl carbazide solution. Make up both solutions to 100 ml each. Wait for 10 to 15 min for the full colour to develop and then find out the percentage transmittance on a photoelectric colorimeter using 460 nm green filter and calculate the corresponding optical density. From the standard graph (*see A-9.2.4*) find out the corresponding concentration of chromium.

### **A-9.3.4 Preparation of Standard Graph**

Weigh 0.283 g of dried potassium dichromate, dissolve in a little water and dilute to 1 l. One millilitre of this solution is equivalent to 0.1 mg chromium (as Cr). Further, dilute 10 ml of the above solution to 1 l. One millilitre of the diluted solution is equivalent to 1 microgram of chromium. Take various volumes of the standard solution so as to give 5, 10, 15, 20 and 25 micrograms of chromium. Add 15 ml aluminium solution to each dilute to about 50 ml and add 30 ml 1 : 1 sulphuric acid and 2 ml bromine water. Further, add 30 percent sodium hydroxide dropwise till the colour disappears and add 2 drops in excess. Boil for 5 min and cautiously add 1 : 1 sulphuric acid till the bromine colour reappears and add 0.5 ml acid in excess. Again boil for about 20 min until bromine colour disappears. Check for bromine, cool and add 1 ml diphenyl carbazide solution and make up the volume to 100 ml. Let it stand for 15 min and then take the reading on a photoelectric colorimeter using green 460 nm filter and a glass cell with 5 cm light path. Find out the corresponding optical density. For adjusting 100 percent transmittance use 50 ml water and proceed in the same manner as above. Plot the optical densities against the corresponding chromium concentrations.

#### A-9.3.5 Calculation

$$\text{Chromium, parts per million, in the sample solution} = \frac{\alpha}{1000}$$

$$\text{therefore, chromium } (X_2), \text{ parts per million, in the salt} = 3.125 \times \frac{\alpha}{1000}$$

where,

a = mass in micrograms of chromium contained in 1 l of the sample.

### A-9.4 Estimation of Molybdenum

#### A-9.4.1 Principle

Molybdenum is extracted as the 8-hydroxyquinoline complex and then converted into the sulphate, which is further reacted with alkali thiocyanate in the presence of a strong reducing agent like stannous chloride, to give a cherry red colour. The colour is extracted with an organic solvent and the intensity is measured in a photoelectric colorimeter.

#### A-9.4.2 Reagents

**A-9.4.2.1 Anhydrous potassium pyrosulphate.**

**A-9.4.2.2 Dilute sulphuric acid** — 10 percent (v/v).

**A-9.4.2.3 Ferrous ammonium sulphate** — 1 percent.

**A-9.4.2.4 Potassium thiocyanate** — 5 percent.

**A-9.4.2.5 Stannous chloride 10 percent.**

Dissolve 10 g stannous chloride in 20 ml 1:1 hydrochloric acid and dilute to 100 ml. Filter, if not clear.

**A-9.4.2.6 Mixture of isoamyl alcohol and carbon tetrachloride** — 1:1.

**A-9.4.2.7 Sodium molybdate** — AR grade.

#### A-9.4.3 Procedure

Extract 2 litres of the sample solution (*see A-2.3*) with 8-hydroxyquinoline by the method described in **A-9.2.4**. Evaporate chloroform completely in a well ventilated fume chamber and ignite at low temperature. Add 5 g of potassium pyrosulphate and fuse in a muffle furnace at 850°C, swirling the dish at intervals to spread the melt along the sides of the dish. Add sufficient quantity of dilute sulphuric acid to dissolve the melt and transfer to a separating funnel which has been dried in an oven. Add 1 ml ferrous ammonium sulphate solution, 10 ml of 5 percent potassium thiocyanate solution and mix thoroughly. Add 10 ml of 10 percent stannous chloride solution and make the solution to about 50 ml.

Shake this solution with 20 ml portions of isoamyl alcohol and carbon tetrachloride mixture, collect the alcoholic layer into a 50 ml standard volumetric flask and make up the volume to 50 ml. The extract should be clear. If turbid keep the flask in hot water for a few seconds. Carry out the blank using 1:1 mixture of isoamyl alcohol and carbon tetrachloride and adjust 100 percent transmittance on the photoelectric colorimeter, using a 5 cm light path cell and 460 nm filter. From the standard graph (*see A-9.4.4*) find out the molybdenum concentration.

#### **A-9.4.4 Preparation of Standard Graph**

Dissolve 0.282 g sodium molybdate in 1000 ml water. Further, dilute 10 ml of the above solution to 1000 ml. One millilitre of this solution is equivalent to 1 microgram of molybdenum. Take aliquot parts of the standard molybdenum solution so as to obtain 5, 10, 15, 20 and 25 micrograms of molybdeum. Add 5 ml of dilute sulphuric acid and dilute to about 40 ml. Add 1 ml ferrous ammonium sulphate, 10 ml potassium thiocyanate solution and 10 ml stannous chloride solution. Transfer to a separating funnel and extract with two 20 ml portions of 1 : 1 mixture of isoamyl alcohol and carbon tetrachloride. Combine extracts and make up to 50 ml with the same mixture. Find out the percentage transmittance using a photoelectric colorimeter with green 460 nm filter and a glass cell of 5 cm light path. Find out the corresponding optical density. Plot the optical densities against the molybdenum concentration on a graph paper.

#### **A-9.4.5 Calculation**

$$\text{Molybdenum content in the sample solution, parts per million} = \frac{\alpha}{2000}$$

$$\text{Therefore, molybdeum content ( } X_3 \text{ ) in the salt, parts per million} = 3.125 \times \frac{\alpha}{2000}$$

where,

a = mass in micrograms of molybdenum contained in 2 l of the sample.

**A-9.4.6** Calculate vanadium + Chromium + molybdenum as below:

(Vanadium + Chromium + Molybdenum), =  $X_1 + X_2 + X_3$  parts per million in the salt

### **A-10 TEST FOR ALUMINIUM**

#### **A-10.1 Principle**

Aluminium forms red coloured lake with aluminor reagent. It is then colorimetrically estimated.

#### **A-10.2 Reagents**

##### **A-10.2.1 Buffer Solution**

Dissolve 154 g of ammonium acetate and 6 ml or acetic acid in water and dilute to 1 l.

**A-10.2.2** *Aluminon Reagent* — 0.10 percent aqueous solution. If the solution is reddish, add a drop of ammonia and make it golden yellow.

**A-10.2.3** *Thioglycolic Acid* — 10 percent.

**A-10.2.4** *Potassium Aluminium Sulphate*.

**A-10.2.6** *Potassium Chloride Solution* — saturated.

### **A-10.3 Procedure**

Take 50 ml of the sample solution (*see A-2.3*), acidify with hydrochloric acid, boil and filter. Neutralize the filtrate with ammonia to pH 6.8-7.2. Add 20 ml buffer solution, 1 ml thioglycolic acid solution and 1 ml aluminon reagent. Heat gently on a water-bath at 100°C for exactly 10 min, cool and make up to 100 ml.

For blank, take 50 ml water and follow the above procedure. Adjust 100 percent transmittance with blank using a photoelectric colorimeter with green 525 nm filter and a 5 cm light path glass cell. Find out the percentage transmittance of the sample and the corresponding optical density and from the standard graph (*see A-10.4*) find out the corresponding aluminium content in micrograms.

### **A-10.4 Preparation of Standard Graph**

Dissolve 1.757 g potassium aluminium sulphate in water and make up to 1 l. Dilute 10 ml of the above solution to 1 l. One millilitre of this solution contains 1 microgram of aluminium (as Al). Take 50 ml saturated potassium chloride solution in six 250 ml beakers. Use one as a blank. Into the other beakers pipette out aliquots of standard aluminium solution so as to give 5, 10, 15, 20 and 30 micrograms of aluminium, and make up the volume to 70 ml with water. Add 20 ml buffer, 1 ml thioglycolic acid solution and 1 ml aluminon reagent to each beaker. Keep on a water-bath at 100°C for exactly 10 min, cool and make up to 100 ml with water.

Adjust the colorimeter to 100 percent transmittance with the blank on a photoelectric colorimeter using green 525 nm filter and a 5 cm light path cell and find out the percentage transmittance of the solution. Plot the corresponding optical densities against the aluminium concentration.

### **A-10.5 Calculation**

$$\text{Aluminium content, parts per million, in the sample solution} = \frac{\alpha}{50}$$

$$\text{Therefore, aluminium content in the salt sample, parts per million} = 3.125 \times \frac{\alpha}{50}$$

where,

a = mass in micrograms of aluminium in 50 ml of the sample solution.

## **A-11 TEST FOR TITANIUM, COPPER AND NICKEL**

**A-11.1** Each metal is determined separately and the results are added up.

### **A-11.2 Determination of Titanium**

#### **A-11.2.1 Principle**

Co-precipitation of titanium along with aluminium and the colorimetric determination of titanium with tiron (disodium 1, 2, dihydroxy benzene-3, 5 sulphonate).

#### **A-11.2.2** *Reagents*

**A-11.2.2.1** *Double distilled water*

**A-11.2.2.2** *Aluminium solution*

**A-11.2.2.3** *Concentrated hydrochloric acid* — See IS 265.

**A-11.2.2.4** *Ammonia solution* — 20 percent (m/m).

**A-11.2.2.5** *Concentrated sulphuric acid* — See IS 266.

**A-11.2.2.6** *Tiron* — 10 percent solution.

**A-11.2.2.7** *Thioglycollic acid* — 10 percent solution.

**A-11.2.2.8** *Buffer solution* — Dilute 1.6 g of sodium acetate and 60 g acetic acid to 1 l with double distilled water.

**A-11.2.2.9** *Potassium titanium oxalate*.

#### **A-11.2.3** *Procedure*

Take 2 l of the sample solution (*see A-2.3*) and proceed in the same manner as for chromium (*see A-9.3.3*), till the aluminium precipitate is dissolved in 50 ml boiling concentrated sulphuric acid. Cool and make up to 100 ml. Make two parts of 50 ml each.

In both, add 10 ml thioglycollic acid solution. Keep one beaker for the blank. In the other beaker add 5 ml tiron solution. Adjust the pH of the solution in both the beakers to 3-4 by adding ammonia solution. Finally, add 10 ml buffer solution and dilute to 100 ml with double distilled water. Use the blank to adjust 100 percent transmittance and take the percentage transmittance of the sample solution on a photoelectric colorimeter, using blue 410 nm filter and a cell with 5 cm light path. Find out the corresponding optical density and read out the titanium concentration corresponding to the optical density from the standard graph (*see A-11.2.4*)

#### **A-11.2.4** *Preparation of Standard Graph*

Weigh 3.6875 g potassium titanium oxalate into a beaker. Add 15 g ammonium sulphate and 200 ml of concentrated sulphuric acid. Boil for 5 to 10 min, cool and make up to 1 l. Further, dilute 10 ml of this solution to 1000 ml. One millilitre of this solution contains 5 micrograms of titanium. Pipette out aliquots of the standard titanium solution containing 5, 10, 15, 20 and 25 micrograms titanium in 250 ml beakers. To each beaker add 15 ml 1 : 1 sulphuric acid, 15 ml aluminium solution and add 25 ml water. Then add 10 ml thioglycollic acid and 5 ml tiron solution to each beaker. Adjust the pH of the solution to 3-4 with ammonia or sulphuric acid. Finally add 10 ml buffer solution and make up to 100 ml. Find out the percentage transmittance using 410 nm blue filter and a glass cell of 5 cm light path. For adjusting 100 percent transmittance, use 50 ml water, treated in the same manner as above. Find out the corresponding optical densities and plot the titanium concentration against the optical densities.

#### **A-11.2.5** *Calculation*

$$\text{Titanium content, parts per million, in the sample solution} = \frac{\alpha}{1000}$$

$$\text{Therefore, titanium content (X}_1\text{) in the salt, parts per million} = 3.125 \times \frac{\alpha}{1000}$$

where

a = mass in micrograms of titanium in 1 l of sample solution.

### **A-11.3 Determination of Copper**

#### **A-11.3.1 Principle**

Copper forms a complex with sodium diethyl dithiocarbamate solution, which is soluble in chloroform giving a yellow coloured solution which is determined colorimetrically.

#### **A-11.3.2 Reagents**

##### **A-11.3.2.1 Ammonium citrate solution**

Take 210 ml concentrated ammonia solution and 150 ml water in a 500 ml beaker. Add, while stirring, 200 g of citric acid, cool and make the contents slightly alkaline with ammonia. Add 5 ml sodium diethyldithiocarbamate solution and extract with 10 ml portions of chloroform till the chloroform layer becomes water white. Dilute to 500 ml with water.

##### **A-11.3.2.2 Sodium diethyldithiocarbamate solution**

Take 0.1 g of sodium diethyldithiocarbamate in 100 ml volumetric flask, dissolve in water and dilute to 100 ml. Filter, if not clear, and store in a brown bottle. The solution is stable for about a week. It shall be kept at a cool place.

##### **A-11.3.2.3 Ethylene diamine tetra acetate, sodium salt**

Dissolve 20 g of ethylene diamine tetra acetate, sodium salt in 500 ml of water.

##### **A-11.3.2.4 Phenolphthalein indicator.**

##### **A-11.3.2.5 Chloroform — See IS 5296.**

##### **A-11.3.2.6 Ammonia solution — 10 percent.**

##### **A-11.3.2.7 Copper sulphate (CuSO<sub>4</sub>.5H<sub>2</sub>O).**

#### **A-11.3.3 Procedure**

Take 100 ml of the sample solution (*see A-2.3*) in a dry separating funnel. Add 5 ml ammonium citrate, 20 ml EDTA solution and 2 drops of phenolphthalein indicator solution. Shake vigorously and add ammonia solution till the solution in the funnel turns very slightly pink. Further, add 5 ml of sodium diethyldithiocarbamate solution and mix well. Extract the complex with 10 ml portion of chloroform till the chloroform layer becomes water white and make up to 50 ml. Use pure chloroform for 100 percent transmittance adjustment and find out the percentage transmittance of the sample, using a photoelectric colorimeter, with blue, 560 to 600 nm filter and a 5 cm light path glass cell. Find out the corresponding optical density and from the standard graph (*see A-11.3.4*), read the copper concentration.

#### **A-11.3.4 Preparation of Standard Graph**

Dissolve 0.3928 g of copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in a little water. Add 8 ml concentrated hydrochloric acid and dilute to 1 l. One millilitre of this solution contains 0.1 milligram of copper. Further, dilute 10 ml of the above solution to 1000 ml with water. One millilitre of the diluted solution contains 1 microgram of copper. Pipette out aliquots of standard copper solution so as to contain 5, 10, 15, 20 and 25 micrograms of copper. Dilute to about 100 ml with water and follow the procedure described for the sample (*see A-11.3.3*). Plot the optical densities against the copper concentration on a graph paper.

#### A-11.3.5 Calculation

$$\text{Copper content, parts per million, in the sample solution} = \frac{\alpha}{100}$$

$$\text{Therefore, copper content } (X_2) \text{ in the salt, parts per million} = 3.125 \times \frac{\alpha}{100}$$

Where,

a = mass in micrograms of copper in 100 ml of the sample.

#### A-11.4 Estimation of Nickel

**A-10.4.1 Principle** — Nickel forms a red coloured complex with dimethyl glyoxime which is soluble in chloroform. It can then be determined colorimetrically.

#### A-11.4.2 Reagents

**A-11.4.2.1 Sodium citrate solution** — 10 percent.

**A-11.4.2.2 Dilute ammonia solution** — 1 : 20 (v/v)

**A-11.4.2.3 Dimethyl glyoxime reagent** – 1 percent solution in ethanol.

**A-11.4.2.4 Chloroform.**

**A-11.4.2.5 Hydrochloric acid** — 0.5 N.

**A-11.4.2.6 Bromine water.**

**A-11.4.2.7 Ammonium nickel sulphate** [ $(\text{NH}_4)_2\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ].

#### A-11.4.2.8 Procedure

Take 50 ml of the sample solution (*see A-1.3*) in a separating funnel, add 5 ml of sodium citrate solution and make the contents slightly alkaline with ammonia solution. Add 2 ml dimethyl glyoxime solution and extract the complex formed with chloroform using 5 ml quantities three to four times.

Collect the chloroform layers in a separating funnel. Add 5 ml hydrochloric acid and shake vigorously. Draw off the chloroform layer and collect the acid layer in a 50 ml volumetric flask. Add 10 drops of bromine water and let stand for a few minutes. Add liquor ammonia till the bromine colour disappears and add 1 ml in excess. Cool to below 30 °C, add 1 ml dimethyl glyoxime reagent and dilute to 100 ml with water.

For blank, take 50 ml water in a separating funnel, to this add 5 ml hydrochloric acid, 10 drops of bromine water and let it stand for a few minutes. Add concentrated ammonia solution till the bromine colour disappears. Cool to below 30°C, add 1 ml dimethyl glyoxime reagent and make up the volume to 100 ml

with water. Adjust 100 percent transmittance on the photoelectric colorimeter with this solution using a 465 nm filter and a 5 cm light path glass cell. Find out the percentage transmittance of the sample solution and calculate the corresponding optical density. From the standard graph (*see A-11.4.4*) read out the concentration of nickel.

#### A-11.4.2.9 Preparation of Standard Graph

Dissolve 0.5088 g of ammonium nickel sulphate [ $(\text{NH}_4)_2\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ] in a little water and dilute to 1 l. Dilute 10 ml of this solution to 100 ml. One millilitre of this solution contains one microgram of nickel. Pipette out aliquots of the standard nickel solution so as to give 5, 10, 20, 30 and 50 micrograms of nickel. Dilute to 50 ml with water and follow the procedure as given for the sample (*see A-11.4.3*). Plot the optical densities obtained against the concentration of nickel on a graph paper.

#### A-11.4.3 Calculation

$$\text{Nickel content, parts per million, in the sample solution} = \frac{\alpha}{50}$$

$$\text{Therefore, nickel content } (X_3), \text{ parts per million, in the salt} = 3.125 \times \frac{\alpha}{50}$$

where,

a = mass in micrograms of nickel contained in 50 ml of sample.

**A-11.5** Calculate titanium + copper + nickel as below;

$$\begin{aligned} & (\textit{Titanium} + \textit{Copper} + \textit{Nickel}), \\ & \textit{parts per million, in the salt} = X_1 + X_1 + X_3 \end{aligned}$$

## A-12 DETERMINATION OF GRIT

### A-12.1 Procedure

Use the water insoluble residue obtained in **A-5.1**. Brush the residue into a beaker, treat it with a few millilitres of aqua regia and warm gently. Cool and dilute the solution, and filter through a filter paper. Wash the residue on the filter paper free of chloride, dry and ignite the filter paper in a silica crucible; cool and weigh. Brush the ignited residue on a 6.3 micron IS Sieve and weigh the material retained on the sieve.

## A-13 DETERMINATION OF ALKALINITY

### A-13.1 Reagents

**A-13.1.1** *Standard Hydrochloric Acid* — 0.1 N.

**A-13.1.2** *Methyl Orange Indicator* — Dissolve 0.5 g of methyl orange in 100 ml of water.

### A-13.2 Procedure

Dissolve about 25 g of dried sample preserved in **A-2.1** in about 400 ml of water, filter and wash the residue free from soluble salts. Collect the filtrate and washings. Take 100 ml from the filtrate and washings. Add two or three drops of methyl orange indicator. Titrate the solution with standard hydrochloric acid to colour change from orange to pinks. Note down the burette reading.

### A-13.3 Calculation

$$\text{Alkalinity ( as } Na_2CO_3\text{), percent by mass} = \frac{V \times N \times 5.3}{M}$$

Where,

$V$  = volume in ml of standard hydrochloric acid,

$N$  = normality of standard hydrochloric acid, and

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

## A-14 ION CHROMATOGRAPHY FOR SULPHATES

### A-14.1 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-14.2 Equipment

**A-14.2 .1** *Anion guard column* — a protector of the separator column.

**A-14.2 .2** *Anion Separator column* — suitable for selective separation of ions under analysis.

**A-14.2 .3** *Anion Suppressor device*

Anion micromembrane suppressor is used to analyse the data Detector: Conductivity detector.

**A-14.2 .4** *Software* — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

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

### A-14.3 Reagents and Standards

**A-14.3.1** *Glass or polyethylene sample bottles.*

**A-14.3.2** *Distilled water or deionized water free from the anions of interest.*

**A-14.3.3** *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-14.3.4 Micromembrane suppressor solution (0.025 N of sulphuric acid)** — Dilute 2.8 ml of conc. Sulphuric acid in 4 l of water

#### **A-14.4 Standard solutions**

**A-14.4.1 Sulphate** — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

#### **A-14.5 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-14.6 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-14.7 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.

### **A-15 DETERMINATION OF IRON, CALCIUM, MAGNESIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**

#### **A-15.1 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-15.2 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 3. 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.

Additionally, Table 3 lists the most important spectral interferences at the recommended wavelengths for analysis.

**Table 3 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences**

Sl No.	Element	Wavelength (nm)	Approx. Achievable limits		Interfering Elements
			Radial viewing ( $\mu\text{g}$ )	Axial viewing ( $\mu\text{g}$ )	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
ii)	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
iii)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr

### A-15.3 Reagents and Solutions

#### A-15.3.1. Nitric acid (65 percent) Suprapure

#### A-15.3.2 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

Iron, calcium and magnesium in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

#### **A-15.3.3** *Standard solution*

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/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- 15.3.4** **Sample preparation**

Weigh about 2.5 g of the 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-15.3.5** *Reagent Blank Solution*

Place 50 ml of nitric acid and 1000 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-15.4** **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-15.5** **Procedure**

##### **A-15.5.1** *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**A-15.3.5**). 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-15.5.2** 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  $\mu\text{g/ml}$  of the lead (and/or Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury) 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-15.6 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  $\text{mg/kg}$  of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).

## **ANNEX B**

*(Clause 6.1)*

### **SAMPLING OF COMMON SALT FOR CHEMICAL INDUSTRIES**

#### **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** For drawing 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, clean, dry and air-tight 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**

**B-2.1.1** *For Heaps and Wagons* — Each individual heap or wagon shall constitute a separate lot.

##### **B-2.1.2** *For Packages*

**B-2.1.2.1** All the packages in a single consignment of common salt of the same 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 a separate lot.

**B-2.1.2.2** The number of packages (n) to be selected from the lot shall depend upon the size of the lot (N) and shall be in accordance with Table 4.

**Table 4 Number of Packages to be Selected for Sampling**

Lot Size (1) <i>N</i>	No. of Packages to be Selected (2) <i>n</i>
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

**B-2.1.2.3** The packages shall be selected at random from the lot and in order to ensure the randomness of selection, procedure given in IS 4905 may be followed.

**B-2.1.2.4** 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 For Wagons and Lorries**

From each wagon or lorry, draw with an appropriate sampling instrument, several portions of the material from different parts, for example, front, middle and back, and at different depths. Collect about 25 kg of the sample in this way.

#### **B-3.2 For Heaps**

In case of heaps, draw samples from different heights, depths and intervals. The quantity of sample drawn from each height should be proportional to the quantity of salt present in that section. Collect about 25 kg of the sample in this way.

#### **B-3.3 Preparation of Composite Sample**

Spread out the samples collected and mixed as above (*see* **B-3.1** and **B-3.2**) on a level, clean and hard surface. Flatten it out first and then scoop the mass together into a cone. Flatten the heap again and divide

into four portions. Remove the two diagonally opposite quadrants. Mix the remaining two portions well again, form into a cone and repeat this process until finally about 2 kg of the material is left. This shall be called the composite sample. This composite sample shall be divided into three parts, one for the purchaser, another for the supplier and the third shall be used as a referee sample. Transfer the samples to separate, clean and dry sample containers and label with full identification particulars.

\*Methods for random sampling.

### **B-3.4 Packages**

**B-3.4.1** From each of the packages selected according to **B-2.1.2.3**, withdraw portions of about 500 g each from different parts and collect about 1 kg of sample from each package.

**B-3.4.2** Out of these portions, equal quantities of the material shall be taken 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 another for the supplier and the third shall be used as a referee sample.

**B-3.5** The referee test samples consisting of composite samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, and shall be used in case of any dispute between the two.

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

**B-4.1** Tests for the determination of all the characteristics given in Table 1 shall be performed on the composite sample.

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

**B-5.1** The lot shall be considered as conforming to the specification if the test results for the tests carried on the composite test sample satisfy the corresponding requirements given in 4 and Table 1.