

भारतीय मानक बोर्ड  
भारतीय मानक, अनामक - अनामक  
) 296 का चौथा संशोधन (

*Draft Indian Standard*  
**SODIUM CARBONATE, ANHYDROUS — SPECIFICATION**  
(*Fourth Revision of IS 296*)

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

This standard was first formulated in 1951 and subsequently revised in 1965, 1974 and 1986. In this revision, instrumental test methods for the determination of aluminium, arsenic, calcium, chlorides, copper, iron, lead, sulphates, magnesium and have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, Reference clause has been incorporated.

Sodium carbonate, anhydrous, is used in the manufacture of sodium salts and for other special purposes. It is also used as a reagent in analytical chemistry.

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 sodium carbonate, anhydrous.

## 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>
265 : 2021	Hydrochloric acid — Specification ( <i>fifth revision</i> )
266 : 1993	Sulphuric acid — Specification ( <i>third revision</i> )
323 : 2009	Rectified spirit for industrial use — Specification ( <i>second revision</i> )
336 : 2021	Ether
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
2088 : 1983	Methods for determination of arsenic ( <i>second revision</i> )
2317 : 1975	Method for gravimetric determination of sulphates ( <i>first revision</i> )
3025 (Part 2)/ ISO 11885	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> )
4905 : 2015/ ISO 24153: 2009	Random sampling and randomization procedures ( <i>first revision</i> )

## 3 GRADES

**3.1** The material shall be of two grades, namely, pure and analytical reagent.

## 4 REQUIREMENTS

### 4.1 Description

The material shall be in the form of granular or fine white powder, free from foreign matter and visible impurities, and shall be readily soluble in water, forming a clear, colourless solution.

**4.2** The material shall 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 5 of Table 1.

### 4.3 Specific Requirement for Pure Grade Material for the Manufacture of Lead Azide

The pure grade material when used for the manufacture of lead azide shall satisfy all requirements prescribed in col 3 of Table 1, except in respect of its copper content which shall be not more than 0.5 ppm (as Cu).

## 5 PACKING AND MARKING

### 5.1 Packing

**5.1.1** The material of the pure grade shall be packed in air-tight containers with polyethylene lining, or as agreed to between the purchaser and the supplier.

**5.1.2** The material of the analytical reagent grade shall be packed in glass or other suitable containers on which the material has no action.

## **5.2 Marking**

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

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

**5.2.1** Containers for the analytical reagent grade shall be labelled with full analytical data of the characteristics prescribed in col 4 of Table 1.

### **5.2.2 *BIS Certification Marking***

The containers may also be marked with the Standard Mark.

**5.2.2.1** The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 2016* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

**Table 1 Requirements for Carbonate, Anhydrous**  
(Clauses 4.2, 4.3 and 5.2.1)

Sl. No.	Characteristic	Requirement		Method of Test, Ref to Cl No. in Annex A
		Pure Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)
i)	Loss on ignition at 300°C, percent by mass, <i>Max</i>	2.0	1.0	A-2
ii)	Total alkalinity (as Na <sub>2</sub> CO <sub>3</sub> ), percent by mass, <i>Min</i>	99.5	99.9	A-3
iii)	Matter insoluble in water, percent by mass, <i>Max</i>	0.02	0.01	A-4
iv)	Sulphates (as SO <sub>4</sub> ), percent by mass, <i>Max</i>	0.05	0.002 5	A-5 or A-19
v)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.12	0.002	A-6 or A-19
vi)	Iron (as Fe), percent by mass, <i>Max</i>	0.004	0.000 5	A-7 or A-20
vii)	Nitrates (as NO <sub>3</sub> ), percent by mass, <i>Max</i>	—	0.002	A-8 or A-19
viii)	Phosphates (as PO <sub>4</sub> ), percent by mass, <i>Max</i>	—	0.001	A-9
ix)	Silicates (as SiO <sub>2</sub> ), percent by mass, <i>Max</i>	—	0.002 5	A-10
x)	Heavy metals [as lead (Pb)], ppm, <i>Max</i>	10	5	A-11 or A-20
xi)	Copper (as Cu), percent by mass, <i>Max</i>	0.003	—	A-12
xii)	Ammonia (as NH <sub>4</sub> ), ppm, <i>Max</i>	—	1.0	A-13
xiii)	Substances reducing iodine (as I), percent by mass, <i>Max</i>	—	0.005	A-14
xiv)	Arsenic (as As), ppm, <i>Max</i>	3.0	0.2	A-15 or A-20
xv)	Calcium and magnesium (as Ca and Mg), percent by mass, <i>Max</i>	—	0.02	A-16 or A-20
xvi)	Aluminium (as Al), percent by mass, <i>Max</i>	—	0.001	A-17 or A-20
xvii)	Potassium (as K), percent by mass, <i>Max</i>	—	0.005	A-18

NOTE — The results shall be reported on as received basis.

## 6 SAMPLING

**6.1** The method of drawing representative test samples of the material and criteria for conformity shall be as prescribed in Annex B.

ANNEX A  
(Clause 4.2)  
**ANALYSIS OF SODIUM CARBONATE, ANHYDROUS**

**A-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 LOSS ON IGNITION**

**A-2.1 Procedure**

Place about 25 g of the material, accurately weighed, in a porcelain dish, and heat it either in an air-oven or in an electric furnace, or on a sand-bath maintained at 300°C, for 1 h. Cool in a desiccator and weigh. Preserve the ignited material for subsequent tests.

**A-2.2 Calculation**

$$\text{Loss on ignition, percent by mass} = 100 \times \frac{M_1}{M_2}$$

where

$M_1$  = loss in mass in g, and

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

**A-3 DETERMINATION OF TOTAL ALKALINITY**

**A-3.1 Reagents**

**A-3.1.1** *Standard Hydrochloric Acid* — 1 N and 0.1 N.

**A-3.1.2** *Bromophenol Blue Indicator*

Dissolve 0.1 g of bromophenol blue in 100 ml of rectified spirit conforming to IS 323.

**A-3.2 Procedure**

Weigh accurately about 2.75 g of the material obtained in **A-2.1** and dissolve in 50 ml of water. Add slowly from a burette 50 ml of standard hydrochloric acid (1 N) and further titrate with standard hydrochloric acid (0.1 N) using bromophenol blue as indicator. Calculate the total volume in terms of hydrochloric acid (1 N).

**A-3.3 Calculation**

$$\text{Total alkalinity (as Na}_2\text{CO}_3\text{), percent by mass of ignited material} = \frac{5.3 VN}{M}$$

where

$V$  = volume in ml of standard hydrochloric acid (1 N) used in the titration,

$N$  = normality of standard hydrochloric acid, and

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

#### A-4 DETERMINATION OF MATTER INSOLUBLE IN WATER

##### A-4.1 Procedure

Weigh accurately 10 to 20 g of the material, transfer it to a 400 ml beaker, add about 200 ml of freshly boiled water, and boil the resulting solution for about 10 min. Filter through a tared Gooch crucible or a sintered glass crucible (G No. 4), collecting the filtrate in a 1000 ml volumetric flask. Transfer the insoluble matter completely to the filter crucible using small volumes of hot water, then wash the crucible 4 times using 5 ml of water for each washing; collect the washings with the filtrate, dry the residue to constant mass at 105 to 110°C. Preserve the solution for subsequent tests.

##### A-4.2 Calculation

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

where

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

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

#### A-5 DETERMINATION OF SULPHATES

##### A-5.1 For Pure Grade

###### A-5.1.1 Reagent

**A-5.1.1.1** *Concentrated hydrochloric acid* — See IS 265.

###### A-5.1.2 Procedure

###### A-5.1.2.1 Preparation of solution

Weigh accurately 10 g of the material and dissolve in 100 ml of water. Carefully add concentrated hydrochloric acid to make it acidic, and boil to decompose the carbonate. Cool and, if necessary, filter through a folded filter paper. Wash the filter paper thoroughly, collecting the filtrate and washings together.

**A-5.1.2.2** Carry out the determination of sulphates with the solution obtained in **A-5.1.2.1** as directed in IS 2317.

##### A-5.2 Analytical Reagent Grade

**A-5.2.1 Reagents**

**A-5.2.1.1 Dilute hydrochloric acid** — approximately 2 N.

**A-5.2.1.2 Barium chloride solution** — approximately 10 percent.

**A-5.2.1.3 Methyl orange paper**

**A-5.2.1.4 Industrial methylated spirit**

**A-5.2.1.5 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 (as  $\text{SO}_4$ ).

**A-5.2.2 Procedure**

Dissolve 1 g of the material with 20 ml of water in Nessler cylinder and make slightly acidic to methyl orange paper with hydrochloric acid and add 1 ml in excess. Boil off the carbon dioxide, cool, dilute to 40 ml and add 10 ml of industrial methylated spirit. Mix the contents and add 1 ml of barium chloride solution. Shake vigorously and allow to stand for 1 h. Carry out a control test in another Nessler cylinder using 0.25 ml of standard sulphate solution and the same quantities of other reagents in the same total volume.

**A-5.2.2.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity or precipitate produced in the test with the material is not greater than that produced in the control test.

**A-5.3 Alternative Method**

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

**A-6 DETERMINATION OF CHLORIDES****A-6.1 For Pure Grade****A-6.1.1 Reagents**

**A-6.1.1.1 Dilute nitric acid** — approximately 4 N and free from chlorides.

**A-6.1.1.2 Calcium carbonate** — precipitated.

**A-6.1.1.3 Potassium chromate indicator** — 5 percent solution.

**A-6.1.1.4 Standard silver nitrate solution** — 0.01 N.

**A-6.1.2 Procedure**

Dilute the solution reserved in **A-4.1** to 1000 ml. Transfer 10 ml into a conical flask and add dilute nitric acid in small portions to neutralize the carbonate. Avoid a large excess of the acid. Add precipitated calcium carbonate in small portions until a small amount of it remains after thorough shaking. Add 10 drops of

potassium chromate indicator solution. Titrate against standard silver nitrate solution till the reddish brown tinge persists after brisk shaking.

### A-6.1.3 Calculation

$$\text{Chlorides (as Cl), percent by mass} = 354.6 \frac{VN}{M}$$

where

$V$  = volume in ml of standard silver nitrate solution used in the titration,

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

$M$  = mass in g of the material taken for the test in **A-4.1**.

## A-6.2 For Analytical Reagent Grade

### A-6.2.1 Apparatus

**A-6.2.1.1 Nessler cylinders**— 100 ml capacity.

### A-6.2.2 Reagents

**A-6.2.2.1 Dilute nitric acid**— approximately 5 N.

**A-6.2.2.2 Silver nitrate solution** — approximately 5 percent.

### A-6.2.2.3 Standard chloride solution

Dissolve 0.165 g of sodium chloride, dried at  $105 \pm 5^\circ\text{C}$ , in water and make up to 1000 ml. Further dilute 10 ml to 100 ml. One milliliter of the diluted solution contains 0.01 mg of chloride (as Cl).

### A-6.2.3 Procedure

Weigh accurately 1.0 g of the material into a Nessler cylinder, dissolve in 40 ml of water and add 12 ml of dilute nitric acid followed by 1 ml of silver nitrate solution. Dilute the contents to the mark with water. Carry out a control test in a similar manner in another Nessler cylinder using 2 ml of standard chloride solution. Compare the turbidity in the two cylinders after 5 min.

**A-6.2.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced by the material is not greater than that produced in the control test.

## A-6.3 Alternative Method

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

## A-7 DETERMINATION OF IRON

### A-7.1 Apparatus

**A-7.1.1** *Nessler Cylinders* — 50 ml capacity.

**A-7.2** *Reagents*

**A-7.2.1** *Hydrochloric Acid* — approximately 15 percent (m/v).

**A-7.2.2** *Ammonium Persulphate*

**A-7.2.3** *Potassium Thiocyanate Solution* — approximately 5 percent.

**A-7.2.4** *Dilute Sulphuric Acid* — approximately 10 percent (v/v).

**A-7.2.5** *Standard Iron Solution*

Dissolve 0.702 g of ferrous ammonium sulphate [ $\text{FeSO}_4 (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$ ] in 10 ml of dilute sulphuric acid and dilute with water to 1000 ml. Dilute 100 ml of the solution again to 1000 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

**A-7.3** *Procedure*

Weigh accurately 1.000 g of the material and dissolve in about 20 ml of water. Add about 5 ml of hydrochloric acid and 30 mg of ammonium persulphate, and boil to oxidize the iron. Cool and transfer to a Nessler cylinder; add 5 ml of potassium thiocyanate solution, dilute with water to the 50 ml mark and stir well. Carry out a control test in another Nessler cylinder, using 4 ml and 0.5 ml of standard iron solution for pure grade and analytical reagent grade, respectively in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the cylinders after 5 min.

**A-7.3.1** 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 test.

**A-5.3** *Alternative Method*

Iron may alternatively be determined by instrumental test method as prescribed in **A-20**.

**A-8** *DETERMINATION OF NITRATES*

**A-8.1** *Reagents*

**A-8.1.1** *Dilute Sulphuric Acid* — approximately 5 N.

**A-8.1.2** *Concentrated Hydrochloric Acid* — same as in **A-5.1.1.1**.

**A-8.1.3** *Potassium Nitrate Solution* — 0.001 N.

**A-8.1.4** *Concentrated Sulphuric Acid* — See IS 266.

**A-8.1.5** *Standard Indigo Carmine Solution*

Dissolve 0.20 g of indigo carmine in 400 ml of dilute sulphuric acid, add 20 ml of concentrated hydrochloric acid and sufficient dilute sulphuric acid to produce 1000 ml. Standardize the solution so that 10 ml added

to 3.3 ml of potassium nitrate solution is just decolourized on adding 13 ml of concentrated sulphuric acid and heated to boiling. One millilitre of this solution is equivalent to 0.02 mg of nitrate (as  $\text{NO}_3$ ).

### A-8.2 Procedure

Dissolve 1.00 g of the material in 10 ml of dilute sulphuric acid, add 1 ml of standard indigo carmine solution followed by 10 ml of concentrated sulphuric acid and heat to boiling.

**A-8.2.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the blue colour produced in the test with the material does not entirely disappear.

### A-8.3 Alternative Method

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

## A-9 DETERMINATION OF PHOSPHATES

### A-9.1 Apparatus

**A-9.1.1** *Separating Funnels* — 200 to 250 ml capacity.

**A-9.1.2** *pH Meter* — with glass electrode.

NOTE — Alternatively, universal standard indicator paper may be used.

**A-9.1.3** *Nessler Cylinder* — 50 ml capacity.

### A-9.2 Reagents

**A-9.2.1** *Dilute Sulphuric Acid* — approximately 5 percent (by volume),

**A-9.2.2** *Ammonium Molybdate* — analytical reagent.

**A-9.2.3** *Concentrated Hydrochloric Acid* — same as in **A-5.1.1.1**.

**A-9.2.4** *Dilute Hydrochloric Acid* — approximately 10 percent (by volume).

**A-9.2.5** *Ethyl Ether* — See IS 336.

**A-9.2.6** *Stannous Chloride Solution*

Dissolve 2 g of stannous chloride ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) in concentrated hydrochloric acid and dilute to 100 ml with the acid.

**A-9.2.7** *Standard Phosphate Silicate Solution*

Dissolve 1.43 g of potassium dihydrogen phosphate ( $\text{KH}_2\text{PO}_4$ ) and 23.65 g of sodium silicate ( $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ ) in water and make up to 1000 ml. Store this solution in a waxed glass bottle or polyethylene bottle. Dilute 10 ml of this solution to 1000 ml with water immediately before use. One milliliter of this diluted solution is equivalent to 0.01 mg of phosphate (as  $\text{PO}_4$ ) and to 0.05 mg of silica (as  $\text{SiO}_2$ ).

### A-9.3 Procedure

Weigh accurately 1.00 g of the material and dissolve in 50 ml of water in a platinum dish. Digest on the steam-bath for 20 min. Cool, neutralize with dilute sulphuric acid to a *pH* of about 4, and dilute to about 75 ml. Take 1 ml of standard phosphate-silicate solution and make up to about 75 ml. Add 0.5 g of ammonium molybdate to each solution and when it dissolves, adjust the *pH* to 2 by adding dilute hydrochloric acid. Check the *pH* with the *pH* meter or universal *pH* indicator paper. Heat both the solutions to boiling, cool to room temperature, add 10 ml of concentrated hydrochloric acid to each and dilute to 100 ml with water. Transfer the solutions to separating funnels, add 35 ml of ether to each, shake vigorously and allow to separate. Draw off the aqueous phase and reserve it for determination of silica. Wash the ether phase of each by shaking with 10 ml of dilute hydrochloric acid, allow to separate and drain off and discard this aqueous phase. Add 0.2 ml of freshly prepared stannous chloride solution to each ether extract and shake. If the ether extracts are turbid, wash with 10 ml of dilute hydrochloric acid.

**A-9.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

## A-10 DETERMINATION OF SILICATES

**A-10.1 Apparatus** — same as described under **A-9.1**.

### A-10.2 Reagents

**A-10.2.1 Concentrated Hydrochloric Acid** — same as in **A-5.1.1.1**.

**A-10.2.2 Butanol**

**A-10.2.3 Dilute Hydrochloric Acid** — 1 percent (by volume).

**A-10.2.4 Stannous Chloride Solution** — same as in **A-9.2.6**.

### A-10.3 Procedure

Add 10 ml of concentrated hydrochloric acid to the solution reserved for the determination of silica (**A-9.3**) and transfer to separating funnels. Add 40 ml of butanol, shake vigorously, and allow to separate. Draw off and discard the aqueous phase. Wash the butanol three times with 20-ml portions of dilute hydrochloric acid, discarding the washing each time. Dilute each butanol solution to 50 ml, take 20 ml aliquot of the sample solution and 10 ml aliquot of standard solution and dilute each to 50 ml with butanol. Add 0.5 ml of a freshly prepared 2 percent stannous chloride solution to each and shake. If the butanol extracts are turbid, wash with 10 ml of dilute hydrochloric acid.

**A-10.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

## A-11 DETERMINATION OF HEAVY METALS

### A-11.1 Apparatus

**A-11.1.1 Nessler Cylinders** — 50 ml capacity.

### A-11.2 Reagents

**A-11.2.1 Dilute Hydrochloric Acid** — approximately 5 N, and 1:99 (v/v).

**A-11.2.2 *p*-Nitrophenol Indicator Solution** — 0.25 percent.

**A-11.2.3 Dilute Ammonium Hydroxide** — 1 : 99 (v/v).

**A-11.2.4 Hydrogen Sulphide Solution** — saturated.

#### **A-11.2.5 Standard Lead Solution**

Weigh accurately 1.60 g of lead nitrate and add 50 ml of nitric acid of analytical reagent grade. Dissolve in water and make up the solution to 1000 ml mark. From this, pipette out 10 ml of the solution and dilute it with water to 1000 ml. One millilitre of this solution contains 0.01 mg of lead (as Pb).

#### **A-11.3 Procedure**

Dissolve 100 g of the material in 5 ml of dilute hydrochloric acid (5N) and add 20 ml of water. Add 1 drop of *p*-nitrophenol indicator solution and then add, dropwise, dilute ammonium hydroxide until the solution turns yellow. Add dilute hydrochloric acid (1 : 99) dropwise, until the solution becomes colourless and then add 0.5 ml in excess. Transfer to a Nessler cylinder; add 5 ml of hydrogen sulphide solution, dilute to 50 ml mark and shake well. Carry out a control test using 1 ml of standard lead solution for pure grade and 0.5 ml of standard lead solution for analytical reagent grade in place of the material and the same quantities of the other reagents. Compare the colour produced in the two Nessler cylinders.

**A-11.3.1** The limit prescribed in Table 1 shall be taken as not been exceeded if the having intensity of colour produced in the test with the material is not greater than that produced in the control test.

#### **A-11.4 Alternative Method**

Lead may alternatively be determined by instrumental test method as prescribed in **A-20**.

### **A-12 DETERMINATION OF COPPER**

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

**A-12.1.1 Nessler Cylinders** — 100 ml capacity.

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

**A-12.2.1 Copper-Free Water** — Re-distil ordinary distilled water twice in an all-glass apparatus.

**A-12.2.2 Concentrated Sulphuric Acid** — See IS 266.

**A-12.2.3 Ammonium Hydroxide** — 20 percent (m/m).

**A-12.2.4 Litmus Paper**

**A-12.2.5 Gum Arabic Solution**

Dissolve 5 g of gum arabic in 100 ml of copper-free water. Filter, if necessary.

**A-12.2.6** *Sodium Diethyl Dithio Carbamate Solution*

Dissolve 0.1 g of sodium diethyl dithio carbamate in 100 ml of copper-free water. Filter, if necessary. Preserve in an amber-coloured bottle and store away from light. As the solution deteriorates on Storage, it should not be preserved for more than two weeks.

**A-12.2.7** *Concentrated Standard Copper Solution*

Dissolve 0.3928 g of copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in 250 ml of copper-free water, Pipette out exactly 25 ml of this solution and make up to 100 ml mark. One millilitre of this solution is equivalent to 0.1 mg of copper (as Cu).

**A-12.2.8** *Dilute Standard Copper Solution*

Transfer exactly 10 ml of the concentrated standard copper solution and dilute with copper-free water to 100 ml. One millilitre of this solution is equivalent to 0.01 mg of copper (as Cu).

**A-12.3 Procedure**

Weigh accurately 10.00 g of the material in 50 ml of copper-free water. Add concentrated sulphuric acid (about 8 ml drop by drop till evolution of carbon dioxide ceases. Place the beaker in a water-bath and evaporate to dryness. Dissolve the sodium sulphate formed in 50 ml of copper-free water and neutralize with ammonium hydroxide, using litmus paper. Add 2 to 3 ml of excess of ammonium hydroxide and bring the solution to boil. Filter hot to remove all iron which is likely to interfere, if present in large quantities. Transfer the filtered solution to a Nessler cylinder and add 1 ml of gum arabic solution followed by 10 ml each of ammonium hydroxide and sodium diethyl dithio carbamate solution. Carry out a control test using 3 ml of concentrated standard solution in the case of the copper pure grade and 0.5 ml of dilute standard copper solution in the case of the material required for the manufacture of lead azide in place of the material, and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour in the two Nessler cylinders after 5 min.

**A-12.3.1** The limits prescribed for copper (Cu) for the pure grade and for the material required for the manufacture of lead azide shall be taken as not having been exceeded if the colour produced in the test with the material is not deeper than that produced in the appropriate control test.

**A-12.4 DETERMINATION OF COPPER USING INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES) METHOD**

**A-12.4.1** Copper can be determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method as prescribed in IS 3025 (Part 2).

**A-13 DETERMINATION OF AMMONIA****A-13.1 Apparatus**

**A-13.1.1** *Nessler Cylinders* — 50 ml capacity.

**A-13.2 Reagents**

**A-13.2.1** *Concentrated Hydrochloric Acid* — See IS 265.

**A-13.2.2** *Sodium Hydroxide Solution* — approximately 5 N.

**A-13.2.3** *Potassium Iodide* — crystals.

**A-13.2.4** *Mercuric Chloride Solution* — saturated.

**A-13.2.5** *Potassium Hydroxide* — solid.

**A-13.2.6** *Nessler Solution*

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water and add to it slowly, with stirring, mercuric chloride solution until a slight permanent precipitate forms. 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 over-night, decant the clear solution and keep the solution in a bottle closed with a well-fitting rubber stopper.

**A-13.2.7** *Standard Ammonia Solution*

Dissolve 2.97 g of ammonium chloride in water and make up to 1000 ml. Take 10 ml of this and dilute to 1000 ml. One millilitre of this solution contains 0.01 mg of ammonia (as  $\text{NH}_4$ ).

### **A-13.3 Procedure**

Dissolve 5.000 g of the material in 25 ml of water and add 8.5 ml of concentrated hydrochloric acid. Boil to remove carbon dioxide, cool and transfer to a Nessler cylinder and add 2 ml of sodium hydroxide solution and 2 ml of Nessler solution. Dilute the contents to the mark. Carry out a control test, using 0.5 ml of standard ammonia solution, 2 ml of Nessler solution and 2 ml of sodium hydroxide solution in the same total volume of the reaction mixture.

**A-13.3.1** The limit prescribed in Table 1 shall be taken as not been exceeded if the having intensity of the colour produced in the test with the material is not greater than that produced in the control test.

## **A-14 DETERMINATION OF SUBSTANCES REDUCING IODINE**

### **A-14.1 Reagents**

**A-14.1.1** *Starch Solution*

Triturate 5 g of starch and 0.01 g of mercuric iodide with 20 ml of water in a mortar. Pour the resulting paste into 1 l of boiling water, boil for 3 min. Allow the solution to cool and decant off the clear liquid.

**A-14.1.2** *Standard Iodine Solution* — 0.01 N.

**A-14.1.3** *Dilute Hydrochloric Acid* — approximately 5 N.

### **A-14.2 Procedure**

Dissolve 10 g of the material in 100 ml of water. Add 2 ml of starch solution and 20 ml of hydrochloric acid. Titrate the contents with standard iodine solution.

**A-14.2.1** The limit prescribed in Table 1 shall be taken to have not been exceeded if not more than 0.4 ml of iodine is required to produce a permanent blue colour.

## **A-15 DETERMINATION OF ARSENIC**

### **A-15.1 Procedure**

Dissolve 1.0 g of the material for pure grade and 5.0 g for analytical reagent grade in 25 ml of water and neutralize with dilute sulphuric acid, using methyl orange as indicator. Carry out the test for arsenic as directed in IS 2088, using for comparison standard arsenic trioxide solution containing 0.003 96 g and 0.001 32 g of arsenic trioxide for pure and analytical grades, respectively.

### **A-15.2 Alternative Method**

Arsenic may alternatively be determined by instrumental test method as prescribed in **A-20**.

## **A-16 DETERMINATION OF CALCIUM AND MAGNESIUM**

### **A-16.1 Reagents**

**A-16.1.1** *Dilute Hydrochloric Acid* — approximately 5 N.

**A-16.1.2** *Sodium Hydroxide Solution* — approximately 5 N.

#### **A-16.1.3** *Standard Magnesium Solution*

Dissolve 1.013 g of magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in water and dilute to 1000 ml. One millilitre of the solution contains 0.1 mg of magnesium (as Mg).

#### **A-16.1.4** *Borate Buffer Solution*

Dissolve, in water, 40 g of the sodium borate, 10 g of sodium hydroxide and 5 g of sodium sulphate and dilute the solution to one litre.

#### **A-16.1.5** *Eriochrome Black T Indicator Solution*

Dissolve 0.1 g of eriochrome black T in 20 ml of rectified spirit.

#### **A-16.1.6** *Standard Ethylene Diamine Tetra-Acetate (EDTA) Solution*

Dissolve 40 g of disodium ethylene diamine tetra-acetate dihydrate in water and dilute to one litre. Standardize the solution by titrating against standard magnesium solution following the procedure given in **A-16.2** Dilute the solution appropriately to obtain N/100 solution.

### **A-16.2 Procedure**

Dissolve 5.00 g of the material in a mixture of 80 ml of water and 20 ml of dilute hydrochloric acid. Boil for 5 min. Cool and neutralize to pH 7.0 with sodium hydroxide solution (about 1 ml). Add 1 ml of standard magnesium solution and sufficient borate buffer solution (about 2 ml) to adjust pH to 10.0. Add 0.1 ml of

eriochrome black T indicator solution and titrate slowly, while shaking, with standard EDTA solution, using a microburette until a pure blue end point is obtained.

**A-16.2.1** The material shall be taken to have passed the test if not more than 4.25 ml of standard EDTA solution is required for the titration.

### **A-16.3 Alternative Method**

Calcium and magnesium may alternatively be determined by instrumental test method as prescribed in **A-20**.

## **A-17 DETERMINATION OF ALUMINIUM**

### **A-17.1 Apparatus**

**A-17.1.1** *Nessler Cylinders* — 50 ml capacity.

### **A-17.2 Reagents**

**A-17.2.1** *Dilute Acetic Acid*— Dissolve 28.5 g of glacial acetic acid in 100 ml of water.

**A-17.2.2** *Ammonium Aurine Tricarboxylate (Aluminon) Solution*

Dissolve 0.1 g of ammonium aurine tricarboxylate in 100 ml of water.

**A-17.2.3** *Ammonium Carbonate Solution* — approximately 5 N.

**A-17.2.4** *Standard Aluminium Solution*

Dissolve 1.680 g of aluminium ammonium sulphate [ $\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ] and dilute to 1000 ml. Further dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of aluminium (as Al).

**A-17.2.5** *Ammonium Acetate* — solid.

### **A-17.3 Procedure**

Transfer 10 ml of the sample solution (*see A-5.1.2.1*) into a Nessler cylinder. Add 10 ml of dilute acetic acid and 1 ml of ammonium aurine tricarboxylate solution. Allow to stand for 5 min and then add 10 ml of ammonium carbonate solution. Carry out a control test in another Nessler cylinder, using 1.0 ml of standard aluminium solution, 15 ml of water, 1 g of ammonium acetate, 5 ml of dilute acetic acid, 1 ml of ammonium aurine tricarboxylate solution and adding, after a lapse of 5 min, 10 ml of ammonium carbonate solution. Make up the volume in the two cylinders to 50 ml and compare the colour produced in the two cylinders after 5 min.

**A-17.3.1** The limit prescribed for aluminium shall be taken as not having been exceeded if any pink colour produced in the test with the material is not deeper than that produced in the control test.

### **A-17.4 Alternative Method**

Aluminium may alternatively be determined by instrumental test method as prescribed in **A-20**.

## A-18 DETERMINATION OF POTASSIUM

**A-18.1** *Simple Flame Photometer* — equipped with interference filter.

### A-18.2 Reagents

#### A-18.2.1 *Standard Potassium Chloride Solution*

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

#### A-18.2.2 *Calibration Graph*

Take 10, 20, 40, 60 and 80 ml of standard potassium solution (*see A-18.2.1*) and dilute to 100 ml with water in different measuring flasks. Each flask now contains 0.01, 0.02, 0.04, 0.06 and 0.08 g potassium chloride per 100 ml. Use these dilute solutions to obtain a corresponding galvanometer reading as given in the procedure (*see A-18.3*) and plot the concentration against galvanometer reading in a rectangular coordinate 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-18.2.3 *Sample Solution*

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

### A-18.3 Procedure

**A-18.3.1** 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-18.3.2** 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 and 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 water and adjust the pointer to zero in galvanometer scale by zero adjustment knob. Then spray the potassium chloride standard solution (**A-18.2.1**) and adjust the deflection to maximum (100) by using sensitivity control knob. Again spray water to see pointer comes to zero; then spray a standard solution to indicate 100. Repeat till water reads zero and standard solution reads 100 with same adjustment during both the operations. A reading zero by water and with the same adjustment 100 by standard solution indicates that the instrument has been now made ready for measurement.

**A-18.3.3** Without altering the earlier adjustment of the instrument spray various diluted solutions prepared in **A-18.2.1** and obtain a calibration graph in the range 0.01 to 0.1 percent potassium chloride. After washing with 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-18.4 Calculation

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

where

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

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

## A-19 ION CHROMATOGRAPHY FOR CHLORIDES, NITRATES AND SULPHATES

### A-19.1 Principle

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

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

### A-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

**A-19.2.4 Detector** — Conductivity detector

**A-19.2.5 Software**

Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

**A-19.2.6 Sample loop** of 100  $\mu\text{l}$ , 200  $\mu\text{l}$ , 500  $\mu\text{l}$  or 1000  $\mu\text{l}$  be used to determine ionic concentration as per instrument manual and practice.

### A-19.3 Reagents

**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-19.4.1 Chloride** — Dissolve NaCl, 1.6485 g in 1 l of reagent water

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

**A-19.4.3 Nitrate Stock Solution**

Dry approximately 2 g of sodium nitrate ( $\text{NaNO}_3$ ) at 105 °C for 24 h. Dissolve exactly 1.3707 g of the dried salt in reagent water, and dilute to 1 l with reagent water in a volumetric flask.

**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 grams 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. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used-

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

**A-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.

**A-20 DETERMINATION OF ALUMINIUM, ARSENIC, CALCIUM, IRON, LEAD AND MAGNESIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD****A-20.1 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-20.2 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 (µg)	Axial viewing (µg)	
(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)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr

vi)	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
viii)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr

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### A-20.3 Reagents and Solutions

#### A-20.3.1 Nitric acid (65 percent) Suprapure

#### A-20.3.2 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 µg/ml of aluminium, arsenic, calcium, iron, lead and magnesium, in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

#### A-20.3.3 Standard solution

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask & 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-20.3.4 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-20.3.5 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-20.4 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-20.5 Procedure**

#### **A-20.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**A-20.5.2**). 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-20.5.2** 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, Iron, calcium, magnesium, arsenic and aluminium 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-20.6 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).

**SAMPLING OF SODIUM CARBONATE, ANHYDROUS****B-1 GENERAL REQUIREMENTS OF SAMPLING**

**B-1.1** In drawing samples, the following precautions and directions shall be observed.

**B-1.2** Samples shall not be taken in an exposed place.

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

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

**B-1.5** The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

**B-1.6** The sample containers shall be of such a size that they are nearly filled by the sample.

**B-1.7** Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

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

In any consignment of one grade of the material, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment of one grade of the material is known to consist of different batches of manufacture or of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

**B-2.2** For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers ( $n$ ) to be selected for this purpose shall depend on the size of the lot ( $N$ ) and shall be in accordance with Table 3.

**Table 3 Scale of Sampling**  
(Clause B-2.2)

Lot Size	Number of Containers to be Selected
$N$	$n$

(1)	(2)
Up to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

**B-2.3** These containers shall be selected at random from the lot and in order to ensure randomness of selection, random sampling procedures given in IS 4905 may be followed.

### **B-3 TEST SAMPLES AND REFEREE SAMPLE**

#### **B-3.1 Preparation of Test Samples**

**B-3.1.1** Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for the characteristics given in Table 1 and should be about 400 g.

**B-3.1.2** Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 800 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

**B-3.1.3** The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 50g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as a referee sample.

#### **B-3.2 Referee Sample**

The referee sample shall consist of the composite sample (*see* **B-3.1.2**) and a set of individual samples (*see* **B-3.1.3** marked for this) purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

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

**B-4.1** Tests for the determination of chlorides and iron shall be conducted on each of the individual samples analytical reagent grade.

**B-4.2** Tests for all the characteristics for pure grade and analytical reagent grades shall be conducted on the composite sample.

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

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

**B-5.1.1** *For Chlorides*

**B-5.1.1.1** *For analytical reagent grade*

The lot shall be considered to have passed the requirement if each of the individual samples satisfies the test laid down in **A-6.2.3**.

**B-5.1.2** *For Iron*

The lot shall be considered to have passed the requirement if each of the individual samples satisfied the test laid down in **A-7.3**.

**B-5.2 For Composite Sample**

The test results on the composite sample shall meet the requirements specified in Table 1.

**B-5.2.1** A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics listed in Table 1.