

भारतीय मानक मसौदा
सोडियम क्लोराइड, विश्लेषणात्मक अभिकर्मक — विशिष्टि
(IS 4408 का दूसरा पुनरीक्षण)

Draft Indian Standard
**Sodium Chloride, Analytical Reagent —
Specification**
(*Second Revision of IS 4408*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

(*Formal clauses will be added later*)

This standard was first published in 1967 and subsequently revised in 1979. In the first revision, a new requirement of ferrocyanide content was added. Requirements for nitrates, ammonium salts, bromide and iodide and insoluble salts were made more stringent. Limits for sulphate and iron content were suitably modified.

In this revision, instrumental test methods for the determination of nitrates, sulphates, iron, lead, calcium, magnesium and arsenic have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Amendment No. 1 and Reference clause have been incorporated. Further, Packing and Marking clause has been updated.

Sodium chloride is used in argentimetry, colorimetry, flame photometry, polarography and in the preparation of buffer solutions. The material required for such purposes has to be free from all impurities which affect the results of analysis.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Draft Indian Standard
**SODIUM CHLORIDE, ANALYTICAL REAGENT —
SPECIFICATION**
(*Second Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium chloride, analytical reagent grade.

2 REFERENCES

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

3 REQUIREMENTS**3.1 Description**

The material shall be in the form of cubes, white crystals or a crystalline powder. One part of the material dissolves in 2.8 parts of water at 25 °C.

3.2 The material shall also conform to the requirements laid down in Table 1, when tested in accordance with the methods prescribed in Annex B. Reference to the relevant clauses of the Annex is given in col 4 of the table.

Table 1 Requirements for Sodium Chloride, Analytical Reagent
(*Clauses 3.2, 4.2, B-3.2.1, B-4.2.1, B-5.2.1, B-6.2.1, B-7.2.1, B-8.3.1, B-11.3.1, C-4.2 and C-5.5*)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. of Annex B)
(1)	(2)	(3)	(4)
i)	Sodium chloride, percent by mass, <i>Min</i>	99.9	B-2
ii)	Sulphate (as SO ₄), percent by mass, <i>Max</i>	0.002	B-3 or B-18
iii)	Nitrate (as NO ₃), percent by mass, <i>Max</i>	0.000 5	B-4 or B-18
iv)	Phosphates (as PO ₄), percent by mass, <i>Max</i>	0.000 5	B-5
v)	Iron (as Fe), percent by mass, <i>Max</i>	0.000 3	B-6 or B-17
vi)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.000 3	B-7 or B-17
vii)	Barium (as Ba), percent by mass, <i>Max</i>	0.001	B-8
viii)	Calcium and magnesium together (as Ca), percent by mass, <i>Max</i>	0.005	B-9 or B-17
ix)	Potassium (as K), percent by mass, <i>Max</i>	0.005	B-10
x)	Ammonium salts (as NH ₄), percent by mass, <i>Max</i>	0.000 5	B-11
xi)	Arsenic (as As), percent by mass, <i>Max</i>	0.000 05	B-12 or B-17
xii)	pH of 10 percent solution	Between 5.0 and 9.0	B-13
xiii)	Bromide and iodide (as Br), percent by mass, <i>Max</i>	0.005	B-14

xiv)	Insoluble matter, percent by mass, <i>Max</i>	0.003	B-15
xv)	Ferrocyanide [as Fe (CN) ₆] percent by mass, <i>Max</i>	0.000 1	B-16

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in airtight glass containers or as agreed to between the purchaser and the supplier.

4.2 Marking

- a) The containers shall be labelled with the name of the material;
- b) Name of the manufacturer and recognized trade-mark, if any;
- c) Net mass; and
- d) Batch number.

The maximum limits for the characteristics given in col 2 of Table 1 shall also be given on the label.

4.2.1 BIS Certification Marking

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

5 SAMPLING

Representative samples of the material shall be drawn and their criteria for conformity shall be determined in accordance with the method prescribed in Annex C.

ANNEX A

(Clause 2)

(LIST OF REFERRED STANDARDS)

<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 323 : 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 2088 : 2023	Methods for determination of arsenic (<i>second revision</i>)
IS 2214 : 1977	Specification for silver nitrate, pure and analytical reagent (<i>first revision</i>)
IS 3025(Part 2) : 2019/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>)
IS 3025 (Part 65) : 2022	Methods of sampling and test physical and chemical for water and wastewater part 65 application of inductively coupled plasma mass spectrometry (ICP-MS) — determination of selected elements including uranium isotopes (<i>first revision</i>)
IS 4905: 2015/ ISO 24153: 2009	Random sampling and randomization procedures (<i>first revision</i>)

ANNEX B

(Clause 3.2, and Table 1)

METHODS OF TESTS FOR SODIUM CHLORIDE, ANALYTICAL REAGENT

B - 1 QUALITY OF REAGENTS

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

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

B-2 DETERMINATION OF SODIUM CHLORIDE

B-2.1 General

Two methods for determination of sodium chloride are given. Method **B-2.2** shall be the referee method, to be used in case of dispute.

B-2.2 Gravimetric Method

B-2.2.1 Reagents

B-2.2.1.1 Dilute nitric acid — approximately 5 N.

B-2.2.1.2 Silver nitrate solution

Dissolve 42 g of solid silver nitrate, analytical reagent grade (*see* IS 2214) in 1 000 ml of water.

B-2.2.2 Procedure

Weigh accurately about 0.5 g of the material freshly ignited at 500 °C and dissolve in 150 ml of water. Acidify with 5 ml of dilute nitric acid. Add silver nitrate solution slowly with stirring until it is in slight excess. (About 35 ml of silver nitrate solution will be required) Heat to boiling, allow to cool in the dark and then filter through a tared sintered glass crucible (G. No. 4) or Gooch crucible with asbestos bed. Wash the residue with water containing a little nitric acid and then with water, till the washings are free from silver nitrate. Dry the crucible with residue at (130 ± 2) °C in an air-oven with thermostatic control and weigh.

B-2.2.3 Calculation

$$\text{Sodium chloride, percent by mass} = \frac{40.78 \times M_1}{M_2}$$

where

M_1 = mass in g of the precipitate, and

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

B-2.3 Volumetric Method

B-2.3.1 Reagents

B-2.3.1.1 *Potassium chromate indicator solution* — 5 percent.

B-2.3.1.2 *Standard silver nitrate solution* — 0.1 N.

B-2.2.2 Weigh accurately about 5 g of the material freshly ignited at 500 °C and dissolve in water. Make up the volume to 100 ml in a volumetric flask. Transfer 10 ml of the solution 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.

B-2.2.3 Calculation

$$\text{Sodium chloride (as NaCl), percent by mass} = 584.5 \frac{V \times N}{M}$$

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 1 000 ml of solution taken for the test.

B-3 TEST FOR SULPHATES

B-3.1 Reagents

B-3.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

B-3.1.2 *Barium Chloride Solution* — dissolve 122 g of solid barium chloride in 1 000 ml of water.

B-3.2 Procedure

Weigh accurately 5 g of the material and dissolve in 50 ml of water in a tall-form beaker. Add to the solution 1 ml of dilute hydrochloric acid and 1 ml of barium chloride solution. Cover and allow to stand for 24 h.

B-3.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if no turbidity or precipitate is produced.

B-3.3 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed at **B-18**.

B-4 TEST FOR NITRATES

B-4.1 Reagents

B-4.1.1 *Indigo Carmine Solution*

Dissolve 0.1 g of indigo carmine previously dried for 2 h at 105 °C in a mixture of 10 ml of concentrated sulphuric acid and 80 ml of water, and dilute to 100 ml.

B-4.1.2 *Dilute Sulphuric Acid* — approximately 5 N.

B-4.2 Procedure

Weigh accurately 1 g of the material, dissolve in 10 ml of water, and add 1 ml of indigo carmine solution and 10 ml of dilute sulphuric acid. Heat to boiling.

B-4.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the blue colour does not entirely disappear.

B-4.3 Alternative Method

Nitrates may alternatively be determined by instrumental test method as prescribed at **B-18**.

B-5 TEST FOR PHOSPHATES

B-5.1 Reagents

B-5.1.1 *Dilute Sulphuric Acid* — approximately 5 N.

B-5.1.2 *Phosphate Reagent No. 1*

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

B-5.1.3 *Phosphate Reagent No. 2*

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

B-5.1.4 *Standard Phosphate Solution*

Dissolve 1.43 g of potassium dihydrogen ortho-phosphate in water and make up to 1 000 ml in a measuring flask with water. Store in a polyethylene bottle. Dilute 1 ml of this phosphate solution to 100 ml in a measuring flask immediately before use. One millilitre of this diluted solution contains 0.01 mg of PO₄.

B-5.2 Procedure

Dissolve 2 g of accurately weighed material in 20 ml of water, add 3 ml of dilute sulphuric acid, 1 ml of phosphate reagent No. 1 and 1 ml of phosphate reagent No. 2, and heat over water-bath at 60°C for 10 min. Transfer the contents in a 50 ml Nessler cylinder (*A*). Do not wash. Repeat the test with 1 ml of standard phosphate solution and take this solution in another 50 ml Nessler cylinder (*B*).

B-5.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if blue colour produced in *A* is not deeper than that produced in *B*.

B-6 TEST FOR IRON

B-6.1 Reagents

B-6.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

B-6.1.2 *Potassium Permanganate Solution* — approximately 0.1 N.

Dissolve 3.3 g of potassium permanganate in 1 050 ml of water and boil the solution gently in flask for about 25 min. Stopper and allow to stand in the dark for a few days. Decant through glass wool into another bottle, protected from light. Do not wash the residue.

B-6.1.3 *Ammonium Thiocyanate Solution* — approximately 50 percent (*m/v*).

Dissolve 50 g of ammonium thiocyanate in sufficient water and dilute to 100 ml.

B-6.1.4 *Solvent*

Mix equal volumes of iso-amyl alcohol and iso-amyl acetate.

B-6.1.5 *Standard Iron Solution*

Dissolve 0.702 g of ferrous ammonium sulphate [FeSO₄(NH₄)₂SO₄ · 6H₂O] in water containing 10 ml of dilute sulphuric acid (10 percent) and dilute to 1 litre in a volumetric flask. Before use, again dilute 10 ml of the solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.001 mg of iron (as Fe).

B-6.2 Procedure

Dissolve 2 g of the material accurately weighed in 10 ml of water, add 1 ml of dilute hydrochloric acid. Add one drop of potassium permanganate solution and mix. Add 5 ml of ammonium thiocyanate solution and 10 ml of solvent for extraction. Shake vigorously and allow the layers to separate. Carry out a control test using 6 ml of standard iron solution. Match the colour developed in the upper solvent layers.

B-6.2.1 The material shall be taken to have not exceeded the limit prescribed in Table 1 if the intensity of the colour obtained with the material is not greater than that obtained in the control test.

B-6.3 Alternative Method

Iron may alternatively be determined by ICP-OES instrumental test method as prescribed at **B-17** or ICP-MS method as prescribed in IS 3025 (Part 65).

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

B-7 TEST FOR HEAVY METALS

B-7.1 Reagents

B-7.1.1 Acetic Acid — approximately 33 percent (*m/v*).

B-7.1.2 Dilute Ammonium Hydroxide Solution — approximately 10 percent (*v/v*).

B-7.1.3 Concentrated Nitric Acid — see IS 264.

B-7.1.4 Standard Lead Solution

Dissolve 1.60 g of lead nitrate [Pb (NO₃)₂] in water and make up the solution to 1 000 ml with water. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0.01 mg of lead (as Pb). Final dilution shall be done immediately before the test.

B-7.1.5 Sodium Sulphide Solution — approximately 10 percent (*m/v*).

B-7.2 Procedure

Weigh accurately 2 g of the material and dissolve in 45 ml of water, and add 5 ml of acetic acid. Transfer the solution to a 100 ml Nessler cylinder. Make it alkaline to litmus by adding slowly dilute ammonium hydroxide solution. Carry out a control test in another 100 ml Nessler cylinder in the same way using 1 ml of standard lead solution. Add 2 drops of sodium sulphide solution to both the solutions and mix thoroughly. Compare the colours developed in both the cylinders.

B-7.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

B-7.3 Alternative Method

Lead may alternatively be determined by ICP-OES instrumental test method as prescribed at **B-17** or ICP-MS method as prescribed in IS 3025 (Part 65).

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

B-8 TEST FOR BARIUM

B-8.1 Apparatus

B-8.1.1 Nessler Cylinders — 100 ml capacity.

B-8.2 Reagents

B-8.2.1 Dilute Hydrochloric Acid — 1 N.

B-8.2.2 Barium Chloride Solution — approximately 10 percent (*m/v*).

B-8.2.3 Dilute Sulphuric Acid — 5 N.

B-8.2.4 Rectified Spirit — see IS 323.

B-8.2.5 Standard Sulphate Solution

Dissolve 0.148 g of ignited sodium sulphate (Na₂SO₄) in water and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of sulphate (as SO₄).

B-8.2.6 Sodium Thiosulphate Solution — approximately 0.1 N.

B-8.3 Procedure

Dissolve 7 g of the material in 40 ml of water and add 10 ml of rectified spirit; mix, add 0.5 ml of dilute sulphuric acid, mix immediately and set aside for 1 h. Discharge any colour due to liberated iodine by the dropwise addition of 0.1 N sodium thiosulphate solution. To another Nessler cylinder add 0.5 ml of standard

sulphate solution, 30 ml of water, 10 ml of rectified spirit and 1 ml of dilute hydrochloric acid, mix and add 1 ml of barium chloride solution, mix immediately and allow to stand for 1 h.

B-8.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced with the material is not greater than that produced with the standard solution.

B-9 TEST FOR CALCIUM AND MAGNESIUM

B-9.1 Reagents

B-9.1.1 Standard Calcium Solution

Weigh 1g of calcium carbonate dried at 120 °C and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 litre in a volumetric flask. One millilitre of the solution is equivalent to 0.400 8 mg of calcium (as Ca).

B-9.1.2 Standard EDTA Solution

Dissolve 3.72 of disodium ethylene diamine tetra-acetate dihydrate in water and dilute in a volumetric flask to 1 litre. The solution shall be standardized frequently against the standard calcium solution, following the procedure given in **B-9.2**.

B-9.1.3 Eriochrome Black T Indicator Solution

Dissolve 0.1 g of the dye in 20 ml of rectified spirit (*see* IS 323). This solution shall be prepared fresh every week.

B-9.1.4 Ammonium Chloride-Ammonium Hydroxide Buffer Solution

Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (relative density 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 litre.

NOTE — Five millilitres of the 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.

B-9.2 Procedure

B-9.2.1 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.

B-9.2.2 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 **B-9.2.1** and note the final titration value. Calculate the calcium equivalent of 1 ml of EDTA solution (say *A*).

B-9.2.3 Dissolve 10 g of the material in 100 ml of water and transfer to a 250 ml conical flask. Add 20 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 solution changes to pure blue end point. Note the volume of EDTA solution used in the titration.

B-9.3 Calculation

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

where

A = calcium equivalent in mg of 1 ml of EDTA solution, determined in **B-9.2.2**,

*V*₁ = volume in ml of standard EDTA solution used in **B-9.2.3**, and

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

B-9.4 Alternative Method

Calcium and magnesium may alternatively be determined by ICP-OES instrumental test method as prescribed at **B-17** or ICP-MS method as prescribed in IS 3025 (Part 65).

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

B-10 TEST FOR POTASSIUM

B-10.1 Flame Photometer — equipped with interference filter.

B-10.2 Reagents**B-10.2.1 Standard Potassium Solution**

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

B-10.2.2 Calibration Graph

Take 10 ml, 20 ml, 40 ml, 60 ml and 80 ml of standard potassium solution (*see B-10.2.1*) and dilute to 1 000 ml with water in different measuring flasks. Each flask now contains 0.01 g, 0.002 g, 0.004 g, 0.006 g and 0.008 g potassium chloride per 100 ml. Use these dilute solutions to obtain a corresponding galvanometer reading as given in **B-10.3.1** and plot the concentrations against galvanometer readings in a rectangular co-ordination graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.001 to 0.01 percent potassium chloride.

B-10.2.3 Sample Solution

Dissolve approximately 20 g of the sample in minimum quantity of water and dilute to 100 ml in measuring flask with water.

B-10.3 Procedure

B-10.3.1 Use a flame photometer equipped with atomizer, burner, optical selective device consisting of 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.

B-10.3.2 Insert the potassium filter corresponding to wavelength 767 nm, light the burner fed by illuminating gas (laboratory gas) adjust the specified air pressure between 0.5 Kg/cm² to 0.6 Kg/cm² and maintain the above air pressure constant such that flame is non-luminous, by turning the control knob. First spray distilled water and adjust the pointer to zero in galvanometer scale by zero adjustment knob. Then spray the potassium chloride standard solution (**B-10.2.1**) and adjust the deflection to maximum 100 by using sensitivity control knob. Again spray water to see that the 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 indicate that the instrument has been made ready for measurement.

B-10.3.3 Without altering the earlier adjustment of the instrument spray various diluted solution prepared in **B-10.2.2** and obtain a calibration graph in the range 0.001 to 0.01 percent potassium chloride. After washing with distilled water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of potassium chloride in the solution (say A).

B-10.4 Calculation

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

where

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

M = mass of dried sample taken for the test.

B-11 TEST FOR AMMONIUM COMPOUNDS**B-11.1 Apparatus**

B-11.1.1 Nessler Cylinders — 50 ml capacity.

B-11.2 Reagents

B-11.2.1 Sodium Hydroxide Solution —10 percent.

B-11.2.2 Nessler Solution

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly, while stirring, saturated mercuric chloride solution until a slight permanent precipitate is formed. Add 30 g of potassium

hydroxide and, when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia free water. Allow to settle overnight, decant the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

B-11.2.3 Standard Ammonium Chloride Solution

Dissolve 0.296 g of ammonium chloride (NH_4Cl) in water and dilute to 1 000 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of this diluted solution is equivalent to 0.001 mg of ammonium (as NH_4).

B-11.3 Procedure

Dissolve 2 g of the material in 45 ml of water and add 15 ml of sodium hydroxide solution. Filter through a sintered glass crucible, previously washed with sodium hydroxide solution. Take 6 ml of the filtrate, dilute with water to 50 ml and add 2 ml of Nessler solution. In a control test, having an equal volume of the solution and containing 1 ml of standard ammonium chloride solution and 1.5 ml of sodium hydroxide solution, add 2 ml of Nessler solution.

B-11.3.1 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.

B-12 TEST FOR ARSENIC

B-12.1 Procedure

Weigh accurately 1 g of the material and dissolve in 50 ml of water. Carry out the test for arsenic as described in IS 2088 using 0.000 660 mg of arsenic trioxide (As_2O_3) for preparing the comparison stain.

B-12.2 Alternative Method

Arsenic may alternatively be determined by ICP-OES instrumental test method as prescribed at **B-17** or ICP-MS method as prescribed in IS 3025 (Part 65).

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

B-13 TEST FOR pH OF 10 PERCENT SOLUTION

B-13.1 Apparatus

B-13.1.1 pH Meter — equipped with a standard calomel electrode and a glass electrode.

B-13.2 Procedure

Dissolve 10 g of the material in 100 ml of water that is free of carbon dioxide and ammonia. Determine pH of the solution by means of the pH meter.

B-14 TEST FOR BROMIDE AND IODIDE

B-14.1 Reagents

B-14.1.1 Anhydrous Disodium Hydrogen Orthophosphate

B-14.1.2 Sodium Hypochlorite Solution

Free from bromide and iodide and containing 2 percent available chlorine.

B-14.1.3 Sodium Formate

B-14.1.4 Potassium Iodide — solid.

B-14.1.5 Ammonium Molybdate Solution — 10 percent (*m/v*).

B-14.1.6 Dilute Sulphuric Acid — 5 N.

B-14.1.7 Standard Sodium Thiosulphate Solution — 0.02 N.

B-14.2 Procedure

Dissolve 15 g of the material and 1 g of anhydrous disodium hydrogen orthophosphate in 45 ml of water and add 5 ml of sodium hypochlorite solution. Heat to boiling for 1 min, add a solution of 2.5 g of sodium formate in 5 ml of water, boil again for 5 min, cool, and dilute to 100 ml with water. Add 1 g of potassium iodide, 30 ml of dilute sulphuric acid and 1 drop of ammonium molybdate solution. Titrate with standard sodium thiosulphate solution using starch solution as indicator.

B-14.2.1 The material shall be taken to have passed the test if not more than 2.8 ml of the standard sodium thiosulphate is required in the titration.

B-15 TEST FOR INSOLUBLE MATTER

B-15.1 Procedure

Weigh accurately about 20 g of the material and dissolve in 200 ml of water, heat to boiling, and digest in a covered beaker on the steam-bath for 1 h. Filter through Whatman filter paper No. 40 or a tared Gooch crucible, wash thoroughly with hot water (about 60 °C) and dry at (105 ± 2) °C. Cool and weigh till constant mass is obtained.

B-15.2 Calculation

$$\text{Insoluble matter, percent by mass} = 100 \frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

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

B-16 TEST FOR FERROCYANIDE

B-16.1 Reagents

B-16.1.1 *Dilute Sulphuric Acid* — approximately 5 N.

B-16.1.2 *Iron Reagent*

Dissolve 10 g of ammonium ferrous sulphate and 1 g of ammonium ferric sulphate in a mixture of 20 ml of water and 4 ml of dilute sulphuric acid; dilute with water to 100 ml.

B-16.1.3 *Phosphate Reagent*

Dissolve 20 g of sodium dihydrogen orthophosphate in 100 ml of water and add 1 ml of sulphuric acid.

B-16.1.4 *Standard Ferrocyanide Solution*

Dissolve 0.2 g of potassium ferrocyanide in water and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of ferrocyanide [as $\text{Fe}(\text{CN})_6$]. Dilute 5 ml of this solution to 100 ml with water immediately before use. One millilitre of this diluted solution is equal to 0.005 mg of ferrocyanide [as $\text{Fe}(\text{CN})_6$].

B-16.2 Procedure

Dissolve 3.5 g of the material accurately weighed in 10 ml of water, add 0.2 ml of dilute sulphuric acid and 0.2 ml of iron reagent, mix and allow to stand for 2 min. Add 1 ml of phosphate reagent, mix again and allow to stand for 5 min. Similarly repeat the above procedure by treating 1 g of the material and 0.5 ml of standard ferrocyanide solution.

B-16.2.1 The material shall be taken to have passed the test if the intensity of blue colour produced with the material is not deeper than that obtained with standard ferrocyanide solution.

B-17 DETERMINATION OF LEAD, IRON, CALCIUM, MAGNESIUM AND ARSENIC BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

B-17.1 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.

B-17.2 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.

Additionally, Table 2 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(Clauses B-17.2 and B-17.4)

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

B-17.3 Reagents and Solutions

B-17.3.1. Nitric Acid (65 percent) Suprapure

B-17.3.2 Standard Stock Solution

Either Prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1 000 µg/ml of Lead, Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

B-17.3.3 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.

B-17.3.4 *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.

B-17.3.5 *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, polytetrafluoroethylene (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.

B-17.4 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.

B-17.5 Procedure

B-17.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**B-17.3.5**). 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.

B-17.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in µg/ml of the lead (and/or Iron, calcium, arsenic) in the sample solution.

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

B-17.6 Calculation

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

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

B-18 ION CHROMATOGRAPHY FOR NITRATES AND SULPHATES

B-18.1 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.

B-18.2 Equipment

B-18.2.1 Anion Guard Column — a protector of the separator column

B-18.2.2 Anion Separator Column — suitable for selective separation of ions under analysis.

B-18.2.3 Anion Suppressor Device

Anion micromembrane suppressor is used to analyse the data.

B-18.2.4 Detector — conductivity detector

B-18.2.5 Software

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

B-18.2.6 Sample loop of 100µl, 200µl, 500µl or 1 000µl be used to determine ionic concentration as per instrument manual and practice.

B-18.3 Reagents

B-18.3.1 Glass or Polyethylene Sample Bottles.

B-18.3.2 Distilled Water or Deionized Water free from the Anions of Interest.

B-18.3.3 Eluent

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

B-18.3.4 Micromembrane Suppressor Solution — (0.025 N of sulphuric acid)

Dilute 2.8 ml of concentrated Sulphuric acid in 4 litres of water

B-18.4 Standard solutions

B-18.4.1 Sulphate — dissolve 1.81 g of potassium sulphate in 1 litre of reagent water.

B-18.4.2 Nitrate Stock Solution

Dry approximately 2 g of sodium nitrate (NaNO₃) at 105 °C for 24 h. Dissolve exactly 1.370 7 g of the dried salt in reagent water, and dilute to 1 litre with reagent water in a volumetric flask.

B-18.5 Calibration and Standardization

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

B-18.6 Procedure

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

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 µ membrane. This membrane can be directly

attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

B-18.7 Data analysis and Calculations

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

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

ANNEX C

(Clause 5)

SAMPLING OF SODIUM CHLORIDE, ANALYTICAL REAGENT

C-1 GENERAL REQUIREMENTS OF SAMPLING

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

C-1.2 Samples shall not be taken at a place exposed to weather.

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

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

C-1.5 The samples shall be placed in suitable dry and airtight containers.

C-1.6 Each sample container shall be sealed airtight after filling and shall be marked with Fill details of sampling, the date of sampling and the year of manufacture of the material.

C-2 SCALE OF SAMPLING

C-2.1 Lot

All the containers in a single consignment of sodium chloride, analytical reagent grade, drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

C-2.2 For ascertaining the conformity of material in the lot to the requirements of the specification, samples shall be tested from each lot separately.

C-2.3 The number of containers (n) to be selected from the lot shall depend upon the size of the lot (N) and shall be in accordance with Table 3.

Table 3 Number of Containers to be selected

(Clause C-2.3)

Lot Size	Number of Containers to be Selected
N	n
(1)	(2)
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

C-2.4 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, reference may be made to IS 4905. In the absence of random number tables, the following procedure may be adopted.

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

C-3 PREPARATION OF TEST SAMPLES

C-3.1 From each of the n containers selected according to **C-2.3** a representative portion of about 100 g of the material shall be drawn.

C-3.2 Out of these portions equal quantities of the material shall be taken and mixed thoroughly to form a composite sample of about 300 g. The composite sample shall be divided into three parts, one for the purchaser, and another for the supplier and the third used as a referee sample.

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

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

C-3.5 The referee test samples consisting of a composite sample and a set of n individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of any dispute between the two.

C-4 NUMBER OF TESTS

C-4.1 Tests for the determination of sodium chloride content shall be performed on each of the individual samples.

C-4.2 Tests for the determination of all the other characteristics given in Table 1 shall be performed on the composite sample.

C-5 CRITERIA FOR CONFORMITY

C-5.1 For Individual Samples

From the test results, the average (\bar{X}) for the sodium chloride content and the range (R) of test results shall be computed (range being defined as the difference between the maximum and minimum values of test results).

The lot shall be declared to have satisfied the requirement for this characteristic if the expression ($\bar{X}-0.4R$) is greater than or equal to 99.9.

C-5.2 For Composite Sample

The lot shall be considered to have passed in respect of the characteristics tested on the composite test sample if the test results satisfy the corresponding requirements given in Table 1.

C-5.3 The lot shall be considered as conforming to the specification if it satisfies all the criteria given in **C-5.1** and **C-5.2**.