

भारतीय मानक मसौदा
ग्लौबर नमक (सोडियम सल्फेट, डेकाहाइड्रेट), तकनीकी —
विशिष्टि

(आईएस 256 का दूसरा पुनरीक्षण)

Draft Indian Standard
**GLAUBER SALT (SODIUM SULPHATE,
DECAHYDRATE), TECHNICAL— SPECIFICATION**
(*Second Revision of IS 256*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

(*formal clauses to be added later*)

This standard was originally published in 1950 and subsequently revised in 1972. In the first revision, the minimum limit of sodium sulphate content was specified as 98.5 percent. The maximum limit for zinc was incorporated as the product from rayon industry might contain zinc to the extent of 0.4 percent. Changes were made in the sampling procedure and some of the methods of test.

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

Glauber salt is used in dyestuff, paper and pulp, glass, textile, leather and metallurgical industries. In glass industry, it is employed to replace sodium carbonate in the manufacture of plate and sheet partially glass. In textile industry, it is added to dyebath for cotton and wool fabrics to promote even distribution of dye of common salt in curing hides. In leather industry, it is used in place of metallurgical industry, it is used in separation of nickel from copper. Cans of Glauber salt are used for stabilizing the temperature inside thermally insulated packages for shipping. It is also used as a purgative both for human and veterinary purposes.

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 Glauber salt [sodium sulphate, decahydrate ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$)], technical.

2 REFERENCE

2.1 The following standards 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 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>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth 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 4905 : 2015	Random sampling and randomization procedures (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of colourless crystals free from visible impurities and almost completely soluble in water. It has tendency to effloresce in hot climate.

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

Table 1 Requirements for Glauber Salt (Sodium Sulphate, Decahydrate), Technical
(Clauses 3.2, A-5.3.1 and B-5.1.1)

Sl. No.	Characteristic	Requirement	Method of Test (Ref to C.L. No. in Annex A)
(1)	(2)	(3)	(4)
i)	Matter insoluble in water, percent by mass, <i>Max</i>	0.10	A-3
ii)	Sodium sulphate (as Na_2SO_4), percent by mass, <i>Min</i>	98.5	A-4
iii)	Iron (as Fe), percent by mass, <i>Max</i>	0.007	A-5 or A-9
iv)	Zinc (as ZnSO_4), percent by mass, <i>Max</i>	0.40	A-6
v)	Chloride (as NaCl), percent by mass, <i>Max</i>	0.15	A-7 or A-10
vi)	pH (of aqueous solution)	3.5 to 4.0	A-8

Note — The requirements from Sl No. (ii) to (v) are on dry basis.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier.

4.2 Marking

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

- a) Name and grade (technical) of the material;
- b) Net weight of contents;
- c) Year of manufacture;
- d) Manufacturer's name and/or his recognized trade-mark, if any; and
- e) Batch number to enable the lot of manufacture to be traced from records.

4.2.1 BIS Certification Marking

The packages may also be marked with the Standard Mark.

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

5 SAMPLING

5.1 The method of drawing representative samples of the material from a lot, number of tests to be performed and the method of finding out the criteria of conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A (Clause 3.2)

METHODS OF TEST FOR GLAUBER SALT (SODIUM SULPHATE, DECAHYDRATE, TECHNICAL

A-1 QUALITY OF REAGENTS

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

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

A-2 PREPARATION OF SAMPLES

A-2.1 Procedure

To obtain the prepared sample, crush 200 g of the material to pass 1.00 mm IS Sieve. Dry it at 105 ± 2 °C to constant mass. Keep the prepared sample in a clean glass-stoppered weighing bottle for test purposes.

A-3 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure

Weigh accurately about 10 g of the prepared sample. Dissolve in 100 ml of water by warming, if necessary, and by stirring the solution well. Filter through a tared filter paper on sintered glass crucible (G No. 4) or Gooch crucible. Wash the residue thoroughly several times with water till it is free from all soluble compounds and dry at 105 to 110 °C to constant mass.

A-3.2 Calculation

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

where

M_1 = mass in g of the residue, and

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

A-4 DETERMINATION OF SODIUM SULPHATE

A-4.1 Reagents

A-4.1.1 Dilute Hydrochloric Acid — 5 N.

A-4.1.2 Barium Chloride Solution — approximately 10 percent (*m/v*).

A-4.2 Procedure

Weigh accurately about 2 g of the prepared sample and dissolve in about 100 ml of water. Filter to remove undissolved matter and wash the filter paper thoroughly. Take the filtrate and washings in a 250 ml volumetric flask and make up the volume to 250 ml. Pipette out 25 ml in a beaker. Add about 200 ml of water and 10 ml of dilute hydrochloric acid and boil. Add to the hot solution a slight excess of hot barium chloride solution and continue boiling for 2 min to obtain a granular precipitate of barium sulphate. Let it stand for 4 h and filter through a tared sintered-glass crucible (G No. 4) or tared Gooch crucible. Wash the precipitate thoroughly with water till it is free from chlorides, and heat to constant mass at 105 to 110 °C.

A-4.2.1 Excess of barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution by addition of barium chloride in a slow stream with stirring minimizes mechanical occlusion of barium chloride and gives a coarse precipitate, which is less soluble in acids.

A-4.3 Calculation

Sodium sulphate, decahydrate content, percent by mass = $\frac{100 \times 1.3804 \times M_1}{M}$

where

M_1 = mass in g of the dried precipitate, and

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

NOTE — Deduct sulphate equivalent to zinc sulphate as obtained in A-6.

A-5 DETERMINATION OF IRON

5.1 Apparatus

A-5.1.1 Nessler Cylinders — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 Concentrated Hydrochloric Acid — see IS 265.

A-5.2.2 Ammonium Persulphate — solid.

A-5.2.3 Butanolic Potassium Thiocyanate

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

A-5.2.4 Standard Iron Solution

Weigh 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] and dissolve in 10 ml of dilute sulphuric acid (10 percent *v/v*). Dilute with water and make up to 1000 ml in a volumetric flask. Transfer 10 ml of this solution and again dilute with water to exactly 100 ml. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-5.3 Procedure

Dissolve 1.000 g of the prepared sample in 10 ml of water. Transfer to a Nessler cylinder and add 1 ml of hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate. Shake vigorously for 30 min and allow the liquids to separate. Carry out a control test in another Nessler cylinder with 7 ml of standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour of the butanol layer in the two cylinders.

A-5.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour of the butanol layer in the test with the material is not deeper than that produced in the control test.

A-5.4 Alternative Method

Iron may alternatively be determined by instrumental test method as prescribed at **A-9**.

A-6 DETERMINATION OF ZINC**A-6.1 Reagents**

A-6.1.1 Concentrated Nitric Acid — see IS 264.

A-6.1.2 Ammonium Chloride Solution — 20 percent (*m/v*).

A-6.1.3 Concentrated Ammonium Hydroxide — 20 percent (*m/m*).

A-6.1.4 Ammonium Chloride — Ammonium Hydroxide Buffer Solution pH 10.

Dissolve 67.5 g of ammonium chloride in 570 ml of concentrated ammonium hydroxide and dilute to 1 litre with water.

A-6.1.5 Ammonium Fluoride — solid.

A 6.1.6 Mixed Indicator

Dissolve 0.125 g of dimethyl yellow and 0.50 g of eriochrome black T in 100 ml of alcohol. The colour change would be from brown to bottle green.

A-6.1.7 Standard Calcium Solution — 0.01 M.

Dissolve 1.0 g of calcium carbonate in minimum amount of hydrochloric acid and make up the volume with water to 1 litre in a volumetric flask.

A-6.1.8 Standard Disodium Ethylenediaminetetra Acetate (EDTA) Solution – 0.01 M.

Dissolve 3.72 g of disodium salt of ethylenediaminetetra acetate in water. Make up the volume to 1 litre in a volumetric flask and standardize it as in **A-6.1.8.1**.

A-6.1.8.1 Standardization of EDTA solution

Pipette out 25 ml of standard calcium solution in a conical flask. Add 5 ml of ammonium hydroxide and 5 ml of buffer solution and titrate against EDTA solution, using 4 drops of mixed indicator. The colour change is from brown to bottle green.

$$\text{Molarity of EDTA solution} = \frac{25 \times M_1}{V_1}$$

where

M_1 = molarity of standard calcium solution, and

V_1 = volume in ml of EDTA solution used in titration.

A-6.2 Procedure

Weigh accurately about 5 g of the prepared sample and transfer to a beaker. Dissolve in 25 ml of water and add 1 ml of concentrated nitric acid. Heat to boiling and precipitate iron as hydroxide by the addition of 25 ml of ammonium chloride solution and 10 ml of ammonium hydroxide. Allow the precipitate to settle and filter through filter paper (Whatman No. 41 or equivalent). Give three or four washings with warm water. Collect the filtrate. If the volume of the filtrate exceeds 150 ml, concentrate it to 150 ml. To this, add 4 ml of buffer solution and 3 g of ammonium fluoride and titrate against standard EDTA solution, using 4 drops of mixed indicator. The change of colour is from brown to bottle green.

Note — Calcium and magnesium are not likely to be present.

A-6.3 Calculation

$$\text{Zinc (as ZnSO}_4\text{), percent by mass} = \frac{16.14 \times V_2 \times M_2}{M}$$

where

V_2 = volume in ml of standard EDTA solution used in the titration,

M_2 = molarity of EDTA solution, and

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

A-7 DETERMINATION OF CHLORIDES

A-7.1 Reagents

A-7.1.1 Dilute Nitric Acid — approximately 5 N.

A-7.1.2 Sodium Bicarbonate — solid.

A-7.1.3 Potassium Chromate Solution — approximately 5 percent (m/v).

A-7.1.4 Standard Silver Nitrate Solution — 0.1 N.

A-7.2 Procedure

Weigh accurately about 5 g of the prepared sample and dissolve in about 60 ml of water. Filter the residue, if any, through a folded filter paper and wash thoroughly with water, collecting the filtrate and washings in a 250 ml conical flask. Just acidify with dilute nitric acid. Neutralize the excess of the acid by the addition of sodium bicarbonate. Add 1 ml of potassium chromate solution and titrate with standard silver nitrate solution with vigorous shaking until the

first permanent colour deviation from the pure yellow of the suspension is obtained. Carry out a blank titration, using the same quantities of other reagents in the same total volume of the reaction mixture.

A-7.3 Calculation

$$\text{Chlorides (as NaCl), percent by mass} = \frac{5.846 \times N (V_1 - V_2)}{M}$$

where

N = normality of the standard silver nitrate solution,

V_1 = volume in ml of standard silver nitrate solution required for titration,

V_2 = volume in ml of standard silver nitrate solution required for blank, and

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

A-7.4 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed at **A-10**.

A-8 DETERMINATION OF pH

A-8.1 Procedure

Dissolve 10.0 g of the material in 100 ml of freshly boiled and cooled water. Determine pH of the solution by means of a pH meter using glass and calomel electrodes.

A-9 DETERMINATION OF IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-9.1 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-9.2 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 A-9.2 and A-9.4)*

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (μg)	Axial viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-

A-9.3 Reagents and Solutions**A-9.3.1. Nitric acid (65 percent) Suprapure****A-9.3.2 Standard stock solution**

Either Prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1000 $\mu\text{g}/\text{ml}$ of Lead, Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-9.3.3 Standard solution

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

A- 9.3.4 Sample preparation

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

NOTE — Sample should be clear before injecting to the instrument.

A-9.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

A-9.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturer's 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-9.5 Procedure

A-9.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-9.3.5). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

A-9.5.2 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 $\mu\text{g/ml}$ of the Iron in the sample solution.

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

A-9.6 Calculation

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

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

A-10 ION CHROMATOGRAPHY FOR CHLORIDES

A-10.1 Principle

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

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

A-10.2 Equipment

A-10.2.1 *Anion guard column* — a protector of the separator column.

A-10.2.2 *Anion Separator column* — suitable for selective separation of ions under analysis.

A-10.2.3 *Anion Suppressor device*

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

A-10.2.4 *Software*

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

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

A-10.3 Reagents and Standards

A-10.3.1 *Glass or polyethylene sample bottles.*

A-10.3.2 *Distilled water or deionized water free from the anions of interest.*

A-10.3.3 *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 litres of water.

A-10.3.4 *Micromembrane suppressor solution (0.025 N of sulphuric acid)*

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

A-10.4 Standard solutions

A-10.4.1 *Chloride* — Dissolve NaCl, 1.6485 g in 1 litre of reagent water

A-10.5 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-10.6 Procedure

Dissolve between 1 to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1-1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard and deviation of retention force shall not exceed ± 10 percent of RT of calibration. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-10.7 Data analysis and Calculations

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

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

ANNEX B

(Clause 5.1)

SAMPLING OF GLAUBBB SALT (SODIUM SULPHATE, DECAHYDRATE), TECHNICAL**B-1 GENERAL REQUIREMENTS OF SAMPLING**

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

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.5 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**

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot. If the material is received in bulk, every 20 tonnes shall be separated as a lot. A preliminary inspection of the salt for assessing colour and impurities may also be made and this information may be used in forming homogenous

B-2.1.1 Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 3.

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

Lot Size	Number of Containers to be Selected
N (1)	n (2)
Up to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables shall be used. In case such tables are not available, the following procedure may be adopted: Starting from any container, count them as 1, 2, 3 ,....., up to r and so on in a systematic manner, where r is the integral part of N/n . Every r^{th} container thus counted shall be taken out for drawing samples.

B-2.4 The sampling operations for bulk material may preferably be carried out when it is unloaded from lorries or other vehicles.

B-3 TEST SAMPLES AND REFEREE SAMPLES

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 all the characteristics given in **3** and shall not exceed 1 kg.

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 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, and another for the supplier and the third to be used as referee sample (*See IS 4905*).

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 weighing not less than 100 g. 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 in **B-1.4**. 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 referee sample.

B-3.2 When sampling ng from bulk quantity, at least five gross samples, each weighing 1 kg may be collected from each lot. If the material is received in a number of lorries or other vehicles, equal number of gross samples may be collected from each of them by allowing some interval between gross samples. If the sampling has to be carried out from a heap, the five gross samples each weighing 1 kg shall be drawn from different locations on the heap.

B-3.2.1 Individual test samples and composite sample may be prepared from these gross samples in the manner described in **B-3.1.2** and **B-3.1.3**.

B-3.3 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 sodium sulphate shall be conducted on each of the individual samples.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

B-5.1.1 *For Sodium Sulphate*

The test results for sodium sulphate shall be recorded, and the mean and the range for these test results calculated as follows:

Mean (\bar{X}) = sum of the test results divided by the number of test results, and

Range(R) = difference between the maximum and minimum values of test results.

The value of expression ($\bar{X} - 0.6 R$) shall be calculated. If the value of this expression is more than or equal to the limit specified in Table 1, the lot shall be declared to have satisfied the requirements for this characteristic.

B-5.2 For Composite Sample

The test results on the composite sample shall meet the corresponding requirements.

B-5.3 A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics.