

भारतीय मानक बोर्ड  
भारतीय मानक, (अन्योनस) – 255  
(255 नमूना अन्योनस)

*Draft Indian Standard*  
**SODIUM SULPHATE, ANHYDROUS (TECHNICAL GRADE) — SPECIFICATION**  
*(Third Revision of IS 255)*

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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

## FOREWORD

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

Sodium sulphate is obtained as a by product in the manufacture of hydrochloric acid, formic acid, sodium bichromate, and in the rayon industry. It also occurs as a natural deposit.

The first revision of IS 255 was brought out to amalgamate two Indian Standard specifications namely, IS 255 and IS 379 which were withdrawn. Second revision was brought to exclude the pharmaceutical grade which was already, covered by the Indian Pharmacopoeia. Second revision also incorporated Amendment No. 1 to IS 255 issued in December 1968.

In this revision, instrumental test methods for the determination of aluminium, chlorides, chromium and iron have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, Reference clause has been incorporated.

This standard contains clause **5.1** which calls for agreement between purchaser and the supplier.

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 sulphate, anhydrous.

## 2 REFERENCES

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

<i>IS No.</i>	<i>Title</i>
261 : 1982	Specification for copper sulphate ( <i>second revision</i> )
264 : 2005	Nitric acid — Specification ( <i>third revision</i> )
265 : 2021	Hydrochloric acid — Specification ( <i>fifth revision</i> )
299 : 2012	Alumino – ferric — Specification ( <i>fifth revision</i> )
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
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> )

## 3 GRADES

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

*Grade A* — used as a diluent for dyestuffs and detergents, and

*Grade B* — used in paper, glass, chemical and textile industries, and in the manufacture of sodium sulphide.

## 4 REQUIREMENTS

### 4.1 DESCRIPTION

The material shall consist essentially of sodium sulphate, anhydrous. It shall be in the form of a white, free-flowing powder, free from visible impurities and shall be almost completely soluble in water.

**4.2** The material, dried in accordance with the method given in **A-2.1**, shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of the table.

## 5 PACKING AND MAPKING

**5.1 Packing** — The material shall be packed as agreed to between the purchaser and the supplier.

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

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

Table 1 Requirements for Sodium Sulphate, Anhydrous

Sl. No.	Characteristic	Requirement for		Method of Test, (Ref to Cl. No. in Annex A)
		Grade A	Grade B	
(1)	(2)	(3)	(4)	(5)
i)	Sodium sulphate (as Na <sub>2</sub> SO <sub>4</sub> ) , Percent by mass, <i>Min</i>	99.0	98.0	A-3
ii)	Matter insoluble in water, percent by mass, <i>Mass</i>	0.25	0.5	A-4
iii)	Chlorides (as NaCl), percent by mass, <i>Max</i>	0.3	2.0	A-5 or A-10
iv)	Iron, aluminium and chromium (as R <sub>2</sub> O <sub>3</sub> ), percent by mass, <i>Max</i>	0.02	0.02	A-6 or A-11
v)	Iron (as Fe), percent by mass, <i>Max</i>	0.002	0.002	A-7 or A-11
vi)	Loss on drying, percent by mass, <i>Max</i>	0.5	1.0	A-8
vii)	pH (of 10 percent solution )	7.0 to 7.5	7.5 to 8.0	A-9
Note — Sl. No. (i) to (v) are carried out on dry basis.				

**5.2.1 BIS Certification Marking**

The product may also be marked with Standard Mark.

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

**6 SAMPLING**

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

**ANNEX A**  
(Clause 4.2)

**ANALYSIS OF SODIUM SULPHATE, ANHYDROUS****A-1 QUALITY OF REAGENTS**

**A-1.1** Unless specified otherwise, pure chemicals and distilled water (*see* IS 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 SAMPLE**

**A-2.1 Procedure**

Crush 50 g of sample to pass through a 1.00 mm IS Sieve. Dry to constant mass at  $105 \pm 5^{\circ}\text{C}$  to obtain the prepared sample and keep in a clean, glass-stoppered bottle in a desiccator for test purposes.

**A-3 DETERMINATION OF SODIUM SULPHATE (as  $\text{Na}_2\text{SO}_4$ )****A-3.1 Principle**

Sodium sulphate is determined by precipitation with barium chloride solution and weighing as barium sulphate.

**A-3.2 Reagents**

**A-3.2.1 Barium Chloride Solution** — approximately 10 percent.

**A-3.2.2 Dilute Hydrochloric Acid** — approximately 4 N.

**A-3.3 Procedure**

Weigh accurately about 0.2 g of the prepared sample and dissolve in about 100 ml of water. Filter to remove undissolved matter and wash the filter paper thoroughly. To the filtrate and washings add 10 ml of dilute hydrochloric acid and boil. Add slowly 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). Wash the precipitate thoroughly till it is free from chlorides and dry to constant mass at 105 to 110°C.

**A-3.3.1** An excess of barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution by the addition of barium chloride in a slow stream with constant stirring minimizes mechanical occlusion of barium chloride and gives a coarse precipitate which is less soluble in acids.

**A-3.4 Calculation**

Sodium sulphate (as  $\text{Na}_2\text{SO}_4$ ), percent by mass =  $60.86 \times \frac{M_1}{M_2}$

where

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

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

**A-4 DETERMINATION OF MATTER INSOLUBLE IN WATER****A-4.1 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 or sintered crucible (G No. 4) or Gooch crucible. Wash thoroughly the residue till it is free from all soluble compounds and dry at 105 to 110°C to constant mass.

**A-4.2 Calculation**

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

where

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

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

## A-5 DETERMINATION OF CHLORIDES (as NaCl)

### A-5.1 Principle

Chlorides are determined by precipitating with silver nitrate solution and titrating the excess of silver nitrate solution with standard ammonium thiocyanate solution using ferric alum as indicator.

### A-5.2 Reagents

**A-5.2.1** *Standard Silver Nitrate Solution* — 0.1 N.

**A-5.2.1.1** *Concentrated nitric acid* — see IS 261.

**A-5.2.1.2** *Ferric alum indicator solution* — saturated.

**A-5.2.1.3** *Standard ammonium thiocyanate solution* — 0.1 N.

**A-5.2.1.4** *Nitrobenzene*

### A-5.2.1 Procedure

Weigh accurately about 5 g of the prepared sample and dissolve in about 80 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. Add with a pipette 25 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid and 10 ml of nitro benzene. Shake vigorously and add 2 ml of ferric alum indicator solution. Titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change.

### A-5.3 Calculation

$$\text{Chlorides (as NaCl), percent by mass} = \frac{0.5846 (25 - V)}{M}$$

where

$V$  = volume in ml of standard ammonium thiocyanate solution used in the titration, and

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

### A-5.4 Alternative Method

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

## A-6 DETERMINATION OF IRON, ALUMINIUM AND CHROMIUM (as $R_2O_3$ )

### A-6.1 Principle

Iron, aluminium and chromium compounds are determined by precipitation with ammonium hydroxide.

### A-6.2 Reagents

**A-6.2.1** *Ammonium chloride* — solid.

**A-6.2.2 Dilute Ammonium Hydroxide** — approximately 4 N.

**A-6.2.3 Ammonium Nitrate Solution** — approximately 2 percent.

**A-6.2.4 Dilute Hydrochloric Acid** — approximately 4 N.

### **A-6.3 Procedure**

Accurately weigh about 10 g of the prepared sample and dissolve it in about 150 ml of water. Acidify with dilute hydrochloric acid, 2 to 3 drops of concentrated nitric acid, boil for 1 to 2 min, and filter to remove undissolved matter. Wash the filter paper thoroughly and, to the hot solution add, first 3 to 5 g of ammonium chloride and then ammonium hydroxide solution till there is a faint permanent smell of ammonia in order to precipitate completely the iron, aluminum and chromium as hydroxides. Boil for 2 min and filter through Whatman filter paper No. 40 or equivalent. Wash with hot ammonium nitrate solution till free from chlorides. Dry the precipitate at  $100 \pm 2^\circ\text{C}$  and ignite at  $1100^\circ\text{C}$  in a tared crucible to constant mass.

### **A-6.4 Calculation**

Iron, aluminium and chromium (as  $\text{R}_2\text{O}_3$ ), percent by mass =  $\frac{100 M_1}{M_2}$

where

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

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

### **A-6.5 Alternative Method of Chromium**

**A-6.5.1** Follow the method given in **A-12** of IS 299 (but weighing 2 g of sample in water instead of 1 g in case of anhydrous form) as per procedure laid in **A-12.3** of IS 299.

### **A-6.6 Alternative Method of Aluminium and Iron**

Aluminium and iron may alternatively be determined by instrumental test method as prescribed in **A-11**.

## **A-7 DETERMINATION OF IRON (as Fe)**

**A-7.1** Two methods are prescribed, namely:

- a) Calorimetric Method, and
- b) Spectrophotometric Method.

### **A-7.2 Colorimetric Method**

#### **A-7.2.1 Principle**

Iron is determined calorimetrically by visual comparison of the colour in Nessler cylinders, using-potassium thiocyanate.

#### **A-7.2.2 Apparatus**

**A-7.2.2.1 Nessler cylinders** — 50 ml capacity.

#### **A-7.2.3 Reagents**

**A-7.2.3.1** *Concentrated nitric acid* — See IS 264.

**A-7.2.3.2** *Ammonium persulphate* — solid.

**A-7.2.3.3** *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-7.2.3.4** *Dilute sulphuric acid* — approximately 10 percent (v/v).

**A-7.2.3.5** 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. Dilute with water to make up the volume to 1000 ml. Transfer 10 ml of this solution and again dilute with water to make up the volume to 100 ml. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe).

#### **A-7.2.4 Procedure**

Weigh accurately about 1.0 g of the prepared sample, dissolve it in water and make up the volume to 100 ml. Pipette out exactly 10 ml of this solution into a beaker, add 1 ml of nitric acid and boil. Cool, transfer the solution to a Nessler cylinder and add 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 s and allow the liquid to separate. Carry out a control test in the other Nessler cylinder adding slowly from a burette a quantity of the standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture until the colours of butanol layer in the two cylinders are exactly matched.

#### **A-7.2.5 calculation**

$$\text{Iron (as Fe), percent by mass} = \frac{0.01 V}{M}$$

where

$V$  = volume in ml of standard iron solution required in the control test, and

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

### **A-7.3 Spectrophotometric Method**

#### **A-7.3.1 Principle**

Iron is reduced to ferrous state by thioglycolic acid and then treated with excess of ammonium hydroxide. The intensity of the resultant pink coloured complex is measured in a photocolorimeter at 535 nm at pH 10.

#### **A-7.3.2 Apparatus**

**A-7.3.2.1** *Any suitable photoelectric calorimeter.*

#### **A-7.3.3 Reagents**

**A-7.3.3.1** *Dilute sulphuric acid* — 10 percent (v/v).

#### **A-7.3.3.2 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. Dilute with water to make up the volume to 1000 ml. Transfer 10 ml of this solution and

again dilute with water to make up the volume to 100 ml. One ml of this solution is equivalent to 0.01 mg of iron (as Fe).

**A-7.3.3.3** *Concentrated hydrochloric acid* — See IS 265.

**A-7.3.3.4** *Dilute ammonium hydroxide approximately* — 6 N.

**A-7.3.3.5** *Thioglycolic acid (SH.CH<sub>2</sub>COOH) AR* — 10 percent (v/v).

#### **A-7.4 Procedure**

Weigh about 10 g of the sample, dissolve in about 75 ml of water. Add 1 ml of concentrated hydrochloric acid and boil for 2 min. Cool to 15°C and add 10 ml of 10 percent thioglycolic acid solution. Add dilute ammonium hydroxide solution drop by drop till pale pink colour appears keeping the temperature of the solution below 15°C. Add one ml more of dilute ammonium hydroxide solution to bring to pH about 10. Dilute to 100 ml in a standard volumetric flask. Read the intensity of the colour in photoelectric colorimeter using 535 nm as wave length. Note down the reading. Run a blank on all the reagents following the above procedure. Subtract this reading from this reading obtained with the sample. Let this be A.

**A-7.4.1** Prepare a series of standard iron solution by taking 2 ml, 5 ml, 7 ml, 10 ml, 15 ml and 20 ml of standard iron solution. Subject them to above procedure and note down the readings. Plot a standard curve for readings obtained against concentration of iron (as Fe).

#### **A-7.5 Calculation**

$$\text{Iron (as Fe), percent by mass} = \frac{A}{10 M}$$

where

A = mg of Fe from the calibration curve, and

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

#### **A-7.6 Alternative Method**

Iron may alternatively be determined by instrumental test method as prescribed in **A-11**.

### **A-8 DETERMINATION OF LOSS ON DRYING**

#### **A-8.1 Procedure**

Weigh accurately about 5 g of the crushed material into a tared dish and dry at 105 ± 5°C. Cool and weigh and repeat the operation till a constant mass is obtained.

#### **A-8.2 Calculation**

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

where

M<sub>1</sub> = mass in g of the material taken for the test, and

M<sub>2</sub> = mass in g of the residue obtained after drying.



**A-9 DETERMINATION OF pH****A-9.1 Procedure**

Weigh 5 g of the prepared sample and dissolve in 50 ml of water. Determine the pH of the solution with the help of a suitable pH meter, using glass and calomel electrode.

**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 micromembrane 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 l 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 l of water

**A-10.4 Standard solutions**

**A-10.4.1 Chloride** — Dissolve NaCl, 1.6485 g in 1 l 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  $\pm 10$  percent of RT of calibration. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

**A-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.

**A-11 DETERMINATION OF ALUMINIUM AND IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD****A-11.1 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-11.2 Recommended Wavelength, limit of quantification and important spectral interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

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

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing ( $\mu\text{g}$ )	Axial viewing ( $\mu\text{g}$ )	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
ii)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr

### A-11.3 Reagents and Solutions

#### A-11.3.1. Nitric acid (65 percent) Suprapure

#### A-11.3.2 Standard stock solution

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

#### A-11.3.3 Standard solution

Pipette out 5 ml from 100  $\mu\text{g}/\text{ml}$  standard stock solution into a 100 ml volumetric flask & 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- 11.3.4 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-11.3.5 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-11.4 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-11.5 Procedure****A-11.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-11.3.5). 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-11.5.2** 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 Iron and aluminium in the sample solution.

**A-11.6 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).

## ANNEX B

(Clause 6.1)

### SAMPLING OF SODIUM SULPHATE, ANHYDROUS

#### 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 of one grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

**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 col 1 and 2 of Table 3.

Table 3 Number of Containers to be selected

Lot Size $N$ (1)	Number of Containers to be Selected $n$ (2)
3 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  $r^{\text{th}}$  container thus counted shall be taken out for drawing samples.

### **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 all the characteristics given in 4 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, 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 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-1.5**. 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 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 for all the grades.

**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 for the relevant grade 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.