

भारतीय मानक मसौदा
सोडियम फ्लोराइड, तकनीकी — विशिष्टि

(IS 10904 का पहला पुनरीक्षण)

Draft Indian Standard
Sodium Fluoride, Technical — Specification
(*First Revision of IS 10904*)

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ICS 71.040.30

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 9th December 2024

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(*Formal clauses will be added later*)

This standard was first published in 1984. In this revision, instrumental test method for the determination of sulphate has been incorporated. In addition to this, editorial corrections have been made wherever required. Also, Reference clause has been incorporated and Packing & Marking clause has been updated.

Sodium fluoride is one of the important fluorine based chemicals and has wide range of uses. It is used in metal processing for heat treatment of metal and as melting and refining fluxes, in glass and ceramics as fluxing agent and as an opacifier for removing colour due to iron, in cement and bricks as mineralizer in burning of cement and for preventing dusting of calcium silicate bricks, in insecticides (not on living plants and animals) fungicides and rodenticides, as preservative for adhesives and wood, in water fluoridation for dosing public water supply where natural fluorine content is low, in soil treatment to reduce permeability of soil to water, metallurgy (rimmed steel), chemical cleaning, electroplating, dentistry etc.

Sodium fluoride has a melting point of 992 °C and boiling point at 1704 °C. It has a solubility of 4 g per 100 g of water at 0 °C and 5 g per 100 g of water at 100 °C.

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 FLUORIDE, TECHNICAL — SPECIFICATION
(*First Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium fluoride, technical.

2 REFERENCES

The standards listed below 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 revisions, and parties to agreements based on this Indian 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 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 4905 : 2015	Random sampling and randomization procedures (<i>first revision</i>)
ISO 24153 : 2009	

3 REQUIREMENTS**3.1 Description**

The material shall be white crystalline or amorphous powder or granules.

3.2 The material shall also comply with 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 Sodium Fluoride, Technical

(*Clause 3.2, C-4.2, C-5.2*)

SI No	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Sodium fluoride (as NaF), percent by mass, <i>Min</i>	97.0	A-2
ii)	Silica (as Na ₂ SiF ₆), Percent by mass, <i>Max</i>	0.50	A-3
iii)	Sulphate (as Na ₂ SO ₄), percent mass, <i>Max</i>	0.25	A-4 or A-7
iv)	Carbonate (as Na ₂ CO ₃), percent by mass, <i>Max</i>	0.25	A-5
v)	Insoluble matter, percent by mass, <i>Max</i>	0.50	A-6

3.3 Sieve Analysis

When tested according to the method laid down in Annex B, the material shall have sieve analysis as agreed to between the purchaser and the supplier.

4 PACKING AND MARKING**4.1 Packing**

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

4.2 Marking

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

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Batch number;
- d) Gross and net mass; and
- e) Date of manufacture.

4.2.1 BIS Certification Marking

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standard Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the license for use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

Representative test samples of the material shall be prepared and adjudged as prescribed in Annex C.

ANNEX A

(Clause 3.2)

METHODS OF TEST FOR SODIUM FLUORIDE, TECHNICAL

A-1 QUALITY OF REAGENTS

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 DETERMINATION OF SODIUM FLUORIDE

A-2.1 Outline of the Method

Fluorine is separated as H_2SiF_6 by distillation with sulphuric acid, and the distillate is titrated with thorium nitrate solution using sodium alizarin sulphonate as indicator.

A-2.2 Reagents

A-2.2.1 Dilute Sulphuric Acid — approximately 24 N.

A-2.2.2 Thorium Nitrate Solution — approximately 0.05 N.

Dissolve 7.045 g of thorium nitrate tetra hydrate [$\text{Th}(\text{NO}_3)_4 \cdot \text{H}_2\text{O}$] in water and dilute to 1000 ml.

A-2.2.3 Buffer Solution

Dissolve 50 g of monochloroacetic acid in 250 ml of 1 N sodium hydroxide and dilute to 500 ml.

A-2.2.4 Dilute Hydrochloric Acid — approximately 0.1 N.

A-2.2.5 Sodium Hydroxide Solution — 20 g/l (*m/v*).

A-2.2.6 Sodium Alizarin Sulphonate Solution — 0.5 g/l (*m/v*). Dissolve 0.05 g of the indicator in water and dilute to 100 ml.

A-2.2.7 Standard Solution of Sodium Fluoride

Weigh to the nearest 0.01 g about 0.28 g of extra pure anhydrous sodium fluoride, previously heated at 600 °C and cooled in a desiccator. Transfer into the distillation flask containing about 250 mg of quartz powder. Stopper the flask and add through the dropping funnel 35 ml of sulphuric acid solution. Carry out the distillation as described in **A-2.4.3** and collect about 450 ml of distillate in a 500 ml volumetric flask. Dilute to the mark and mix.

A-2.2.7.1 Transfer 50 ml aliquot portion of the distillate in a 250 ml beaker and titrate as described in **A-2.4.5**.

A-2.2.7.2 Carry out a blank test following the same procedure with the same quantities of reagents as described in **A-2.2.7.1** and **A-2.2.7.2**.

A-2.2.7.3 Factor of the standard solution

The mass in g of fluorine (as NaF) corresponding to 1 ml of thorium nitrate solution that is the factor (f) is given by the formula:

$$\text{Factor (f)} = \frac{M_1}{V_1 - V_2}$$

where

M_1 = mass in g of NaF contained in the aliquot portion of the standard solution,

V_1 = volume in ml of the thorium nitrate solution used for titration of the aliquot portion of the standard solution, and

V_2 = volume in ml of the thorium nitrate solution used for titration of aliquot portion of the blank test solution.

A-2.3 Apparatus — Steam distillation assembly in as per Fig. 1.

A-2.4 Procedure

A-2.4.1 Weigh to the nearest 0.01 g about 0.28 g of finely powdered, dried test sample and transfer into the distillation flask containing about 250 mg of quartz powder using 15 ml to 20 ml of water.

Place a 500 ml measuring flask under the condenser to collect the distillate. Connect the distillation flask to the condenser and start water circulation.

A-2.4.2 Stopper the distillation flask and add through the dropping funnel 35 ml of the sulphuric acid solution.

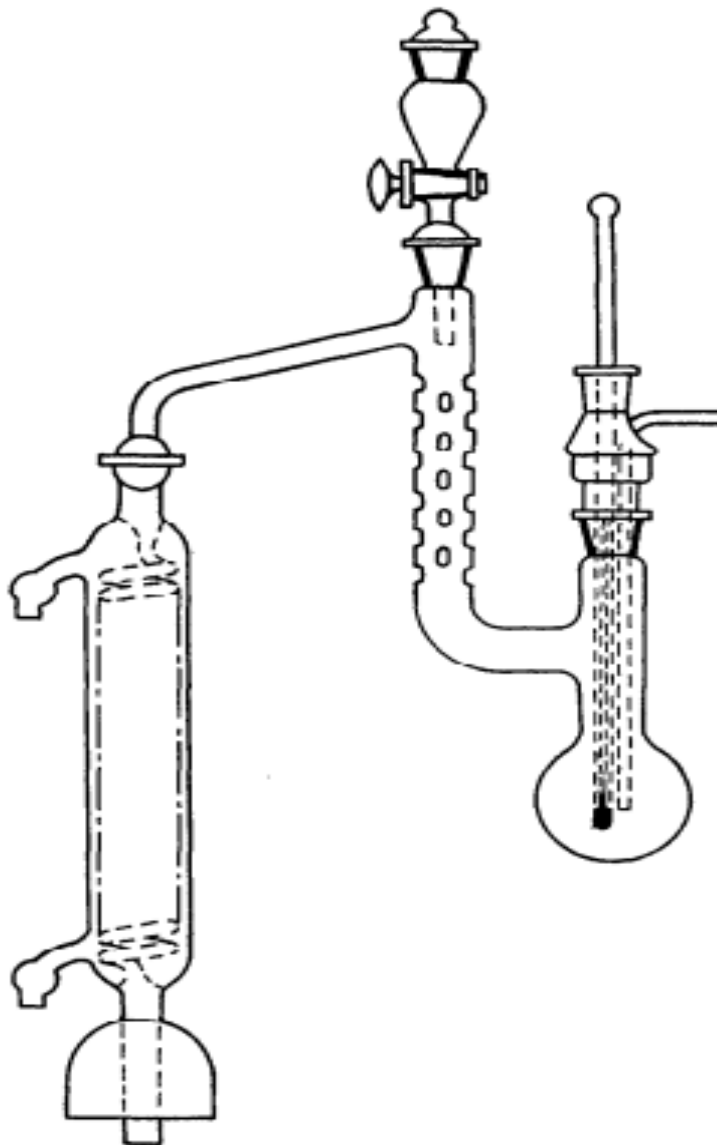


FIG. 1 STEAM DISTILLATION ASS

Fill the steam generating flask, two-third full of water add small pieces of pumice-stone and heat the flask, leaving the steam regulating tube open till the water boils.

A-2.4.3 Heat the distillation flask until the solution reaches 160 °C. Then pass the steam maintaining the temperature of the solution in the distillation flask at (160 ± 2) °C by controlling the heat of the gas burner under the flask. Collect approximately 450 ml of distillate over a period of about 100 min.

A-2.4.4 Disconnect the distillation flask from the steam generator and condenser. Rinse the condenser with a jet of water and add washings to the distillate. Cool to room temperature dilute the distillate to the mark and mix.

A-2.4.5 Transfer 50 ml of the solution obtained in **A-2.4.3** to a 250 ml conical beaker and add 1 ml of sodium alizarin sulphonate solution. Acidify the solution slightly by titration and back-titration with sodium hydroxide solution and hydrochloric acid solution until the reddish tinge formed just disappears. Add 2 ml of the buffer solution and titrate with the thorium nitrate solution until a persistent pale pink colouration is obtained. The Colour becomes particularly noticeable when the precipitated mass is allowed to settle.

A-2.5 Calculation

$$\text{Sodium fluoride as (NaF), percent by mass} = \frac{(V_3 - V_4) \times f}{M} \times 1000$$

where

V_3 = volume in ml of the standard thorium nitrate solution used in titration of aliquot portion of the test solution,

V_4 = volume in ml of the standard thorium nitrate solution used in titration of aliquot portion of blank test solution,

f = factor equivalent to mass in g of sodium fluoride corresponding to 1 ml of the standard thorium nitrate solution (as determined in **A-2.2.7.3**), and

M = mass in g of the test portion.

A-3 DETERMINATION OF SODIUM SILICOFLUORIDE BY COLORIMETRIC METHOD

A-3.1 Outline of the Method

A-3.1.1 Sample is fused with a mixture of sodium carbonate and soda borax. Fused mass is dissolved in nitric acid, treated with ammonium molybdate to form silica-molybdic complex. The complex is reduced in sulphuric acid medium and the Colour intensity of the reduced complex is measured on photoelectric colorimeter at a wavelength of about 630 nm.

A-3.2 Reagents

A-3.2.1 *Sodium Carbonate* — anhydrous.

A-3.2.2 *Borax-Sodium Meta Borate* ($\text{Na}_3\text{B}_4\text{O}_7$) — powder.

A-3.2.3 *Concentrated Nitric Acid* — approximately 65 percent (m/v).

A-3.2.4 *Ammonium Molybdate Solution* — 100 g/l.

Dissolve 25 g of ammonium molybdate tetra hydrate (NH_4)₆Mo₇O₂₄·4H₂O in 200 ml of water at about 50 °C. Allow to cool and transfer to a 250 ml measuring flask. Dilute to the mark, mix and transfer immediately to a polyethylene bottle. Discard the solution when precipitate appears.

A-3.2.5 *Tartaric Acid Solution* — 100 g/l.

A-3.2.6 *Sulphuric Acid Solution* — approximately 16 N.

A-3.2.7 *Reducing Solution*

- Dissolve 0.8 g of sodium sulphite (Na_2SO_3) in 20 ml of water. Add 0.16 g of l-amino, 2-naphthol, 4-sulphonic acid and mix to dissolve.
- Dissolve 10 g of sodium bisulphite ($\text{Na}_2\text{S}_2\text{O}_5$) in 70 ml of water. Mix solution (a) and (b) and dilute to 100 ml and mix. Filter if necessary. Store in an amber colored PE bottle in a cool place.

A-3.2.8 Standard Silica Solution — 0.500 g/l silica (as SiO₂).

Weigh in a platinum crucible to the nearest 0.001 g, 0.500 g of finely powdered pure quartz previously ignited at 1000 °C for 1 h and cooled in a desiccator. Add 5 g of sodium carbonate and mix thoroughly with a platinum rod. Fuse the mass carefully initially at a lower temperature and finally at about 800 °C until a clear transparent melt is obtained. Allow to cool, and little hot water and heat the crucible gently to dissolve the salts. Transfer the solution quantitatively into a PE beaker. Cool, and about 500 ml of water and transfer quantitatively to a 1 000 ml measuring flask. Dilute to the mark and mix. Transfer immediately to a polyethylene bottle. One ml of this solution contains 0.500 mg of silica (as SiO₂).

A-3.2.9 Dilute Standard Silica Solution — 0.010 g/l of silica (as SiO₂).

Transfer 20 ml of silica standard solution (**A-3.2.8**) into a 1000 ml measuring flask, dilute to the mark and mix. Transfer immediately to a polyethylene bottle. One ml of this solution contains 10 micrograms of silica (as SiO₂). This solution should be prepared just before use.

A-3.3 Apparatus

A-3.3.1 Platinum Crucible — 35 ml capacity with lid.

A-3.3.2 Photoelectric Colorimeter — With cells of 2 cm optical path length and with 610 nm filter.

A-3.4 Procedure

A-3.4.1 Weigh 4 g of sodium carbonate and 1.35 g of borax in a platinum crucible, weigh exactly 1 g of finely powdered, dried sample in the same crucible. Mix the contents with a platinum rod.

A-3.4.2 Heat the crucible slowly by keeping in front part of an electric furnace, till the mixture melts. Transfer the crucible in the hottest zone of the furnace at 750 °C to 780 °C temperature and keep it for 30 minutes to complete the fusion. Remove the crucible from the furnace and allow to cool.

A-3.4.3 Place the crucible with lid in a 500 ml polyethylene beaker containing about 150 ml of hot water and 20 ml of nitric acid. Heat the contents on a water bath till the mass dissolves completely. Remove the crucible and lid from the beaker, rinse them with water and add the washings to the solution in the beaker.

A-3.4.4 Cool the solution and transfer quantitatively to a 500 ml measuring flask. Dilute to the mark and mix. Transfer the solution immediately to a polyethylene beaker.

Filter the solution through a dry ordinary filter paper with polyethylene funnel, into a polyethylene bottle.

A-3.4.5 Transfer 50 ml of the test solution into a 100 ml measuring flask, add 5 ml of ammonium molybdate solution, mix and allow to stand for 15 minutes.

NOTE — Here 50 ml aliquot has been suggested, assuming that, sodium fluoride is manufactured from distilled and purified hydrofluoric acid, and there is very little silica in the material. However, the aliquot may be taken depending on the silica content expected.

A-3.4.6 Add 5 ml of tartaric acid solution, 11 ml of sulphuric acid solution and 2 ml of the reducing solution in sequence, stirring the contents after each addition. Dilute to the mark and mix. Allow to stand for 15 minutes.

A-3.4.7 Using the photoelectric colorimeter fitted with a suitable filter, carry out the photometric measurement after adjusting the instrument to zero absorbance against water.

A-3.4.8 Carry out a blank test by the same procedure (**A-3.4.1** to **A-3.4.7**) and the same quantities of all the reagents, but omitting the sample.

A-3.4.9 Preparation of Calibration Graph — Into a series of six 100 ml measuring flasks, transfer, 1, 2, 4, 6, 8, and 10 ml of silica standard solution (**A-3.2.9**).

Then proceed exactly as mentioned in **A-3.4.5** to **A-3.4.7** and measure the absorbency of the standard solutions on the colorimeter.

Plot a graph having the quantities in micrograms of silica (as SiO₂) in the standard solutions as abscissae and the corresponding absorbance values as the ordinates.

A-3.5 Calculation

$$\text{Silica (as Na}_2\text{SiF}_6\text{), percent by mass} = \frac{(M_1 - M_2) \times 3.13}{1\,000 \times M}$$

where

M_1 = mass in micrograms of silica in the test solution,

M_2 = mass in micrograms of silica in the blank test solution, and

M = mass in g of the test sample.

NOTE — Factor for converting silica (as SiO_2 to silica as $(\text{Na}_2\text{SiF}_6)$ is 3.13

A-4 DETERMINATION OF SULPHATE BY GRAVIMETRIC METHOD

A-4.1 Outline of the Method

Sample is fused with sodium carbonate and treated with perchloric acid to convert all sulphate into soluble form and remove fluorine. The residue on evaporation of the solution is dissolved in hydrochloric acid and sulphate is precipitated as BaSO_4 by treatment with barium chloride solution.

A-4.2 Reagents

A-4.2.1 Sodium Carbonate — anhydrous

A-4.2.2 Concentrated Perchloric Acid — 70 percent.

A-4.2.3 Quartz — Fine Powder.

A-4.2.4 Hydrochloric Acid — 36 percent (v/v).

A-4.2.5 Barium Chloride Solution — 100 g/l.

A-4.2.6 Ammonia Solution — relative density 0.91.

A-4.3 Apparatus

A-4.3.1 Platinum Dish — 100 ml capacity.

A-4.4 Procedure

A-4.4.1 Take 8 g of sodium carbonate in the platinum dish and weigh, to the nearest 0.001 g, 2.0 g of the finely powdered dry sample into the same dish. Add 2 g more of sodium carbonate to the dish to cover the sample. Mix with a platinum rod.

A-4.4.2 Place the dish in the front part of the furnace till the mixture melts and then keep it in the control zone at 750 to 780 °C for 30 min to complete the fusion. Remove the dish from the furnace and allow to cool.

A-4.4.3 Add one g of quartz powder and 35 ml of perchloric acid to the dish. Evaporate the mixture to dryness on a sand bath. Allow the dish to cool, add further 30 ml of perchloric acid and evaporate to dryness. Allow to cool.

A-4.4.4 Add 25 ml of hydrochloric acid into the dish and heat to dissolve the salts. Add about 25 ml of water and heat further to boil.

A-4.4.5 Transfer the mixture into a 500 ml measuring flask, add carefully, ammonia solution while stirring the contents, until a litmus paper strip, added to the mixture turns blue. Add 5 ml of ammonia solution more. Cool to room temperature and make up to the mark.

Shake the mixture well and filter through Whatman No. 1 or equivalent filter paper into a 500 ml beaker discarding the first few ml of the filtrate.

A-4.4.6 Transfer 250 ml of the filtrate into a 500 ml glass beaker add few drops of methyl red indicator and then add hydrochloric acid dropwise till the solution turns slightly reddish. Add 2 ml of the acid more and heat the solution to boil. Add 25 ml of hot barium chloride solution in a fine stream with constant stirring, boil the solution for further three minutes and allow to stand for four hours.

A-4.4.7 Filter the hot solution through Whatman No. 42 or equivalent filter paper and wash the precipitate with hot water till free from chloride. Transfer the filter paper with precipitates into a pre weighed porcelain crucible with lid and incinerate at a low temperature. Place the crucible with half covered lid in the furnace and ignite the residue for 20 minutes. Remove the crucible from the furnace, allow to cool and reweigh.

A-4.5 Calculation

$$\text{Sulphate (as Na}_2\text{SO}_4\text{), percent by mass} = \frac{(M_1 - M_2) \times 100}{M} \times 0.6086$$

where

M_2 = mass in g of the residue with the crucible and lid,

M_1 = mass in g of the crucible and lid only, and

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

A-4.6 Alternative Method

Sulphate may alternatively be determined by instrumental test method as prescribed at A-7.

A-5 DETERMINING OF SODIUM CARBONATE BY VOLUMETRIC TITRATION METHOD

A-5.1 Outline of the Method — Sample is titrated with standard hydrochloric acid solution using phenolphthalein as indicator.

A-5.2 Reagents

A-5.2.1 Hydrochloric Acid — 0.1 N.

A-5.2.2 Phenolphthalein Indicator Solution — 10 g/l.

Dissolve 1 g of phenolphthalein powder in 100 ml of ethanol. Add 2 percent sodium hydroxide solution dropwise till very faint pink colouration appears.

A-5.3 Procedure

Weigh to the nearest 0.001 g to 25 g of finely powdered dry sample into a 500 ml beaker. Add about 20 ml of water and 5 drops of phenolphthalein solution. Heat the solution to boil (if the solution turns pink, sodium carbonate is present).

Titrate the solution with standard hydrochloric acid solution to the disappearance of pink Colour.

A-5.4 Calculation

$$\text{Sodium carbonate (as Na}_2\text{CO}_3\text{), Percent by mass} = \frac{V \times N \times 10.6}{M}$$

where

V = volume in ml of hydrochloric acid used in titration,

N = normality of hydrochloric acid, and

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

A-6 DETERMINATION OF WATER INSOLUBLE MATTER

A-6.1 Outline of the Method — Sample is dissolved in water and insoluble matter is separated by filtration, dried and weighed.

A-6.2 Apparatus

A-6.2.1 Filter crucible with sintered glass filter-plate porosity G4 (15 to 30 microns).

A-6.3 Procedure

Weigh to the nearest 0.01 g about 5 g of the finely powdered, dry sample and place it in a 500 ml glass beaker. Add approximately 250 ml of water and heat the solution nearly to boil for about 10 minutes.

A-6.3.1 Allow to cool and immediately filter the solution by vacuum through the pre-weighed filter crucible. Wash with 30 ml portion of water at about 80°C each time for four times. Place the crucible in oven at 110°C and keep for 2 hours. Remove the crucible from oven, allow to cool in a desiccator and weigh.

A-6.4 Calculation

$$\text{Water insoluble matter, percent by mass} = \frac{(M_2 - M_1) \times 100}{M}$$

where

M_2 = mass in g of the crucible containing insoluble matter,

M_1 = mass in g of the empty crucible, and

M = mass in g of the test sample.

A-7 ION CHROMATOGRAPHY FOR SULPHATES

A-7.1 Principle

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

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

A-7.2 Equipment

A-7.2.1 Anion Guard Column — A protector of the separator column.

A-7.2.2 Anion Separator Column — Suitable for selective separation of ions under analysis.

A-7.2.3 Anion Suppressor Device — Anion micro membrane suppressor is used to analyse the data.

A-7.2.4 Detector — Conductivity Detector.

A-7.2.5 Software — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

A-7.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

A-7.3 Reagents

A-7.3.1 *Glass or Polyethylene Sample Bottles.*

A-7.3.2 *Distilled Water or Deionized Water free from the Anions of interest.*

A-7.3.3 *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.

A-7.3.4 *Micro Membrane Suppressor Solution: (0.025 N of sulphuric acid)* — Dilute 2.8 ml of concentrated sulphuric acid in 4 litres of water.

A-7.4 Standard Solutions

A-7.4.1 *Sulphate* — Dissolve 1.81 g of potassium sulphate in 1 000 ml of reagent water.

A-7.5 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.

A-7.6 Procedure

Dissolve between 1g 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.

A-7.7 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 3.3)***METHOD OF SIEVE ANALYSIS FOR SODIUM FLUORIDE****B-1 General**

The material has a tendency to form small globules of very fine particles on the screen while sieving by mechanical device. These small globules do not pass through the screen and give erroneous results about the sieve size. It is therefore recommended to carry out the sieve analysis manually using 4 cm size soft brush.

B-2 APPARATUS

B-2.1 Standard test sieves with 20 cm dia, brass frame resting smoothly on each other and with lid and receiver.

B-3 PROCEDURE

B-3.1 Thoroughly clean the test sieves, with a soft brush and weigh to 1 g on a top-pan balance.

Prepare a next of the sieves, the coarser size sieve, being on top of the finer size sieve with a receiver at the bottom of the next.

B-3.2 Weigh to the nearest 0.1 g to 100 g of the sample and transfer it carefully on the top coarser sieve of the next.

B-3.3 Move the material with a 4 cm size smooth brush, the surface of the screen so that the material travels along the whole surface of the screen in regular frequency. Continue this operation for a period of 15 minutes, stroking the frame of the sieve in between to clear up the apertures that get choked up.

B-3.4 After 15 min. Revive the sieve from the next and transfer the under-size to the top of the next screen. Weigh the screen along with the material and note the net mass of the material retained on the screen.

B-3.5 Follow exactly the same procedure for the material collected on the next screen and note down the net mass of the material that has retained on the screen.

B-3.6 Weigh the receiver together with the material and note the mass of finer material collected in the receiver.

B-4 CALCULATION

B-4.1 The material retained on each sieve as percent by mass is given by the formula;

$$\frac{(M_1 - M_2)}{M} \times 100$$

where

M_1 = mass in g of test sieve and the material,

M_2 = mass in g of the test sieve only, and

M = mass in g of the test sample.

NOTE — Losses of material up to about 1 percent are expected during the procedure, which are distributed to various sizes according to their masses.

ANNEX C*(Clause 4)***SAMPLING OF SODIUM FLUORIDE, TECHNICAL****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 full 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 fluoride, technical, drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different 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 upon the size of the lot (N) and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected

(Clause C-2.3)

Lot Size	Number Of Containers To Be Selected
N	n
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 150 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 cast of any dispute between the two.

C-4 NUMBER OF TESTS

C-4.1 Tests for the determination of sodium fluoride 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 fluoride content and the range (R) of test results shall be computed (range being defined as the difference between the maximum and the minimum values of test results).

The Lot shall be declared to have satisfied the requirement for this characteristic if the expression ($\bar{X} - 0.6 R$) is greater than or equal to 97.0.

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**.