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Indian Standard

SPECIFICATION FOR
STRONTIUM NITRATE FOR
PYROTECHNIC COMPOSITIONS

(First Revision)

UDC 661.843.43 : 662.1

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5 January 1985
AMENDMENT NO. 1 AUGUST 1990
TO
IS 5671 : 1984 SPECIFICATION FOR STRONTIUM
NITRATE FOR PYROTECHNIC COMPOSITIONS

(First Revision)

[Page 4, Table 1, Sl No. (xii), col 2] -
Substitute '[as Sr (NO$_3$)$_2$]' for '[as Sr (NO$_3$)$_3$]'

(CHD 26)

Reprography Unit, BIS, New Delhi, India
Indian Standard
SPECIFICATION FOR
STRONTIUM NITRATE FOR
PYROTECHNIC COMPOSITIONS
( First Revision )

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(Continued on page 2)

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0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 March 1984, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally issued in 1970. The Explosives and Pyrotechnics Sectional Committee responsible for its preparation decided to revise it in the light of experience gained since its publication. In the revised standard, modifications have been made in the requirements of hygroscopicity and calcium content. Alternate instrumental methods have been prescribed for the determination of chlorides and calcium.

0.3 The main use of strontium nitrate is for producing red coloured flame in pyrotechnic compositions. For such uses a material of high degree of purity is required.

0.4 The anhydrous salt is the main type produced commercially. The material is generally produced by (a) treating strontium carbonate with nitric acid, (b) digesting celestite with soda ash followed by treatment with nitric acid, and (c) heating celestite with coal in a rotary kiln to form a soluble sulphide followed by treating with nitric acid. Strontium nitrate solution so formed is purified and commercial anhydrous strontium nitrate crystals of high purity are obtained by crystallization.

0.5 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 : 2-1060*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).
1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for strontium nitrate intended for use in pyrotechnic compositions.

2. REQUIREMENTS

2.1 Description — Strontium nitrate shall be anhydrous in the form of a clean white crystalline powder, free from grit, foreign matter and visible impurities.

2.2 Particle Size — The particle size of the material shall be as agreed to between the purchaser and the supplier.

2.3 Composition — The material shall comply with the requirements laid down in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

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<thead>
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<th>SL No.</th>
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<td>i)</td>
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<td>1.0</td>
<td>A-3</td>
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<td>ii)</td>
<td>Matter insoluble in water, percent by mass, Max</td>
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<td></td>
</tr>
<tr>
<td>a) Total</td>
<td>Max</td>
<td>0.25</td>
<td>A-4</td>
</tr>
<tr>
<td>b) Organic matter</td>
<td>Max</td>
<td>0.1</td>
<td></td>
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<tr>
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<td>Max</td>
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<td></td>
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<tr>
<td>iii)</td>
<td>Hygroscopicity, percent by mass, Max</td>
<td>1.0</td>
<td>A-5</td>
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<td>iv)</td>
<td>$\rho_p$ ( of aqueous solution )</td>
<td>5.5 to 7.5</td>
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<td>v)</td>
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<td>Nil</td>
<td>A-8</td>
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<td>vii)</td>
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<td>Nil</td>
<td>A-9</td>
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<tr>
<td>viii)</td>
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<td>A-10</td>
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<td>ix)</td>
<td>Barium compounds [ as Ba(NO$_3$)$_2$ ], percent by mass, Max</td>
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<td>x)</td>
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<td>xi)</td>
<td>Ammonium compounds ( as NH$_3$ ), percent by mass, Max</td>
<td>0.01</td>
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<tr>
<td>xii)</td>
<td>Strontium nitrate content [ as Sr(NO$_3$)$_3$], percent by mass, Min</td>
<td>97.0</td>
<td>A-14</td>
</tr>
</tbody>
</table>
3. PACKING AND MARKING

3.1 Packing — Strontium nitrate shall be packed in polyethylene lined-jute bags or any other package having an inner layer of polyethylene to prevent ingress of moisture. The type and size of packages shall be subject to agreement between the purchaser and the supplier. However, for transport by rail, the packing shall conform to the provisions of the Indian Railways Conference Association, Red Tariff No. 19.

3.2 Marking — Each package shall be legibly marked with the following information:
   a) Name of the material;
   b) Net mass of the material in the package;
   c) Manufacturer's name and/or his recognized trade-mark, if any;
   d) Year of manufacture; and
   e) Lot number to enable the batch of manufacture to be traced from records.

3.2.1 The packages may also be marked with the ISI Certification Mark.

   NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material from a lot, the number of tests to be performed on each, and the criteria for acceptance shall be as prescribed in Appendix B.
A P P E N D I X A

( Clause 2.3 and Table 1 )

METHODS OF TEST FOR STRONTIUM NITRATE FOR PYROTECHNIC COMPOSITIONS

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water ( see IS : 1070-1977* ) 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 Mix the sample well by rotating the bottle several times and transfer a portion immediately into a wide-mouthed bottle and stopper it. Take care that no pieces of cork or sealing wax get mixed with the sample. Do not expose the sample to an atmosphere containing acid or alkaline fumes.

A-3. DETERMINATION OF MOISTURE

A-3.1 Procedure — Weigh accurately about 5 g of the prepared sample ( see A-2.1 ) in a weighed, clean, dry squat form weighing bottle, and dry to constant mass at 105 ± 2°C. Cool in a desiccator and weigh.

A-3.2 Calculation

\[
\text{Moisture, percent by mass} = 100 \left( \frac{M_2 - M_1}{M} \right)
\]

where

- \( M_2 \) = mass in g of the weighing bottle with the material before drying,
- \( M_1 \) = mass in g of the weighing bottle with the material after drying, and
- \( M \) = mass in g of the material taken for the test.

A-4. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-4.1 Total Insoluble Matter

A-4.1.1 Procedure — Take 5 g of the prepared sample in a 500-ml beaker and dissolve in about 100 ml of water. Filter the residue in a No. 40 Whatman filter paper. Wash the residue with water till free from all soluble matter. Dry in an oven at 105 ± 2°C. Brush out the residue in a weighed silica crucible, and weigh.

*Specification for water for general laboratory use ( second revision ).
A-4.1.2 Calculation

Total matter insoluble in water, percent by mass

\[ \frac{100 \times M_1}{M} \]

where

\( M_1 = \) mass in g of the dried residue, and
\( M = \) mass in g of the material taken for the test.

A-4.2 Organic Matter Insoluble in Water

A-4.2.1 Procedure — Ignite the silica crucible with residue obtained in A-4.1.1 in a muffle furnace at 600 to 700°C. Cool and weigh. The loss in mass is due to the burning of the organic matter.

A-4.2.2 Calculation

Organic matter insoluble in water, percent by mass

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

where

\( M_1 = \) mass in g of the dried residue (see A-4.1.1),
\( M_2 = \) mass in g of the ignited residue, and
\( M = \) mass in g of the sample taken for the test.

A-4.3 Sieve the ignited residue obtained in A-4.2.1 on a 125-micron IS Sieve. The material shall satisfy the requirements of the test if no residue is left on the sieve.

A-5. DETERMINATION OF HYGROSCOPICITY

A-5.1 Procedure — Weigh accurately about 5 g of the dried sample obtained in A-3.1 in a dish of approximately 6 cm diameter and expose it to an atmosphere of 75 percent relative humidity at 27 ± 1°C (see Note) for 24 hours. Weigh again.

NOTE — The test atmosphere of 75 percent relative humidity at 27 ± 1°C may be obtained as follows:

Place in a desiccator of total height not less than 23 cm and internal rim diameter not less than 20 cm an aqueous sludge of sodium chloride. The area of the surface of the sludge should be at least 75 percent of the floor of the desiccator. Supports for keeping the prepared sample in the desiccator shall be so arranged that there is minimum obstruction in the diffusion of water vapour within the desiccator and the height of the exposed prepared sample above the sludge does not exceed 10 cm. The desiccator shall be maintained at a temperature of 27 ± 1°C.
A-5.2 Calculation

Hygroscopicity, percent by mass = \[
\frac{100 \left( M_3 - M_2 \right)}{M_2 - M_1}
\]

where

- \( M_3 \) = mass in g of the dish with the sample after exposure,
- \( M_2 \) = mass in g of the dish with the sample before exposure, and
- \( M_1 \) = mass in g of the empty dish.

A-6. DETERMINATION OF pH

A-6.1 Procedure — Shake 5 g of the powdered sample of size about 150 micron with 100 ml of freshly boiled and cooled water; filter and wash the residue thoroughly with 25 ml of water. Determine the pH of the aqueous extract with pH meter. Make at least two determinations on test samples from two separate portions and if the values differ by more than 0.4, repeat with two fresh portions. Reject the highest and the lowest and report the average. Excessive contact with air shall be avoided.

A-7. DETERMINATION OF WATER SOLUBLE CHLORIDES

A-7.0 General — Two methods are prescribed, namely, volumetric method and automatic chloride titrator method. In case of dispute, volumetric method shall be the referee method.

A-7.1 Volumetric Method

A-7.1.1 Reagents

A-7.1.1.1 Dilute nitric acid — 1 : 4 (v/v), free from lower oxides of nitrogen which give a colour with thiocyanate.

A-7.1.1.2 Standard silver nitrate solution — 0.05 N.

A-7.1.1.3 Standard potassium thiocyanate solution — 0.05 N.

A-7.1.1.4 Ferric alum indicator — 10 percent (m/v).

A-7.1.2 Procedure — Weigh 5 g of the prepared sample and extract in about 100 ml of water. Filter the solution through a No. 40 Whatman filter paper. Wash the residue using about 5 to 10 ml of water. Add to the filtrate 20 ml of dilute nitric acid and a measured excess of silver nitrate solution, usually 20 ml of which is sufficient. If a significant opalescence due to the precipitation of silver chloride is obtained, shake the flask well till all the silver chlorides coagulate and then titrate with a potassium thiocyanate solution using 5 ml of ferric alum indicator. Run a blank determination using all the reagents.
A-7.1.3 Calculation

\[
\text{Chlorides (as NaCl), percent by mass} \quad 100 \times \frac{0.058 \times (X - Y)}{M} \times N
\]

where

- \(X\) = volume in ml of standard potassium thiocyanate solution required for the blank,
- \(T\) = volume in ml of standard potassium thiocyanate solution required for the sample,
- \(N\) = normality of standard potassium thiocyanate solution, and
- \(M\) = mass in g of the prepared sample taken for the test.

A-7.2 Automatic Chloride Titrator Method

A-7.2.1 Reagents

A-7.2.1.1 Nitric acid-acetic acid reagent — To about 800 ml chloride-free double distilled water, add 6.4 ml of concentrated nitric acid (see IS : 264-1976*) and 100 ml of glacial acetic acid (see IS : 695-1975†). Dilute with the same water to 1 litre and mix thoroughly.

A-7.2.1.2 Gelatin reagent — Weigh accurately about 0.62 g of gelatin reagent (60 parts of gelatin, 1 part thymol blue and 1 part thymol) and dissolve in hot water, cool and dilute to 100 ml. Keep always under refrigeration and take out only desired quantity in a test tube.

A-7.2.1.3 Standard sodium chloride solution — Weigh accurately about 0.23 g of dry sodium chloride, dissolve in water and dilute to 100 ml.

A-7.2.2 Procedure - Clean the electrodes of the instrument and rinse with water thoroughly. Weigh accurately 0.1 g to 0.5 g of strontium nitrate sample directly in the titration vessel. Add 4 ml of nitric acid-acetic acid reagent and 0.2 ml gelatin reagent.

A-7.2.2.1 Prepare standard by taking 0.1 ml of standard sodium chloride solution and 4 ml of nitric acid-acetic acid reagent and 0.2 ml of gelatin reagent. Also prepare blank solution.

A-7.2.2.2 Titrate the sample solution, standard solution as prepared in A-7.2.2.1 and the blank according to the instructions of the instrument and record the time.

---

*Specification for nitric acid (second revision).
†Specification for acetic acid (second revision).
A-7.2.3 Calculation

Chlorides (as NaCl), percent by mass

\[
\frac{(B - C) \times M_1 \times 100}{(A - C) \times M}
\]

where

- \( B \) = time in seconds for strontium nitrate sample solution,
- \( C \) = time in seconds for blank,
- \( M_1 \) = mass in g of sodium chloride present in 0.1 ml of standard sodium chloride solution,
- \( A \) = time in seconds for standard solution (A-7.2.2.1), and
- \( M \) = mass in g of strontium nitrate sample taken for the test.

A-8. TEST FOR WATER SOLUBLE CHLORATES

A-8.0 Outline of the Method — Chlorates are tested by comparing the colour produced by the material with aniline hydrochloride.

A-8.1 Reagent

A-8.1.1 Aniline Hydrochloride — Dissolve 18 g of redistilled aniline in 375 ml of concentrated hydrochloric acid and make up the volume to 500 ml with water. Add 2 drops of saturated potassium chlorate solution, shake and allow to stand overnight. Filter off the blue sediment and store the reagent in a glass-stoppered bottle in the dark.

A-8.2 Procedure — Dissolve 5 g of the prepared sample in 40 ml of warm water, filter, if necessary, and make up the volume to 100 ml in a Nessler cylinder with the aniline hydrochloride reagent. Stir with a glass rod for 1 minute to ensure thorough mixing. The material shall be taken to satisfy the requirements of the test if no blue coloration develops in 10 minutes.

A-9. TEST FOR NITRITES

A-9.1 Reagents

A-9.1.1 Sulphanilic Acid Solution — Dissolve 1 g of sulphanilic acid in 100 ml of warm 30 percent \( m/v \) acetic acid.

A-9.1.2 \( \alpha \)-Naphthylamine Solution — Boil 0.3 g of \( \alpha \)-naphthylamine with 70 ml of water, filter off any residue and add 30 ml of glacial acetic acid to the filtrate.

A-9.2 Procedure — Weigh accurately about 25 g of the material, transfer to a 400-ml beaker, add 100 ml of cold water and stir to dissolve. Transfer the solution to a 250-ml volumetric flask and make up the volume with water.
A-9.2.1 Transfer by means of a pipette 10 ml of the solution, prepared as above, to a 50-ml Nessler cylinder, add 2 ml of sulphanilic acid solution and 2 ml of α-naphthylamine solution. Make up to 50-ml mark with water and allow to stand for 15 minutes. The material shall be taken to have satisfied the requirement of the test if no pink colour develops.

A-10. DETERMINATION OF CALCIUM

A-10.0 General — Two methods are prescribed, namely, volumetric method and flame photometric method. In case of dispute, volumetric method shall be the referee method.

A-10.1 Volumetric Method

A-10.1.0 Outline of Method — Calcium is separated from strontium and barium nitrates by treatment with ether-alcohol mixture in which barium and strontium nitrates are insoluble. Calcium is determined by precipitating calcium as oxalate, dissolving the precipitate in dilute sulphuric acid and titrating with standard potassium permanganate solution.

NOTE — Butyl cellosolve may alternatively be used for separation of calcium from barium and strontium instead of ether-alcohol mixture but care should be taken of the toxic hazards of the chemical.

A-10.1.1 Reagents


A-10.1.1.3 Dilute acetic acid — 10 percent (m/v).

A-10.1.1.4 Ammonium oxalate solution — saturated.

A-10.1.1.5 Standard potassium permanganate solution — 0.1 N.

A-10.1.1.6 Dilute sulphuric acid — 10 percent (v/v).

A-10.1.2 Procedure — Take 5.0 g of the sample (see A-2.1) in a 250-ml conical flask and keep inside an electric oven maintained at about 140°C for 2 hours. Cork and cool the flask. Treat the dry residue with 60 ml of absolute alcohol, stopper the flask and allow it to stand with frequent shaking for 1 or 2 hours. Add an equal volume

* Specification for absolute alcohol (revised).
† Specification for ether (second revision).
of anhydrous ether, close the flask, shake and allow it to stand for 12 hours. Filter through Whatman filter paper No. 40 moistened with ether-alcohol mixture (50:50) and wash with ether-alcohol mixture until a few drops of the filtrate evaporated on platinum foil leaves no residue.

A-10.1.2.1 Keep the residue for the estimation of barium and strontium.

A-10.1.2.2 Evaporate the filtrate to dryness on a water-bath away from naked flame at not more than 50°C. Dissolve the residue in 50 ml of water. Add 1 ml of dilute acetic acid, and then 25 ml of ammonium oxalate solution. Boil up to 10 minutes and allow to stand for 2 hours. Filter through a sintered glass crucible No. G4 and wash with cold water until washings are free from oxalates. Transfer the precipitate to a 400-ml beaker containing 50 ml of dilute sulphuric acid. Heat to 80°C and titrate with standard potassium permanganate solution.

10.1.3 Calculation

Calcium compounds, percent by mass = $8.2 \times \frac{V \times N}{M}$

where

$V$ = volume in ml of standard potassium permanganate solution used in the titration,

$N$ = normality of standard potassium permanganate solution, and

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

A-10.2 Flame Photometer

A-10.2.0 General — Calcium content is determined with the help of a flame emission spectrophotometer by measuring the intensity of radiation emitted at 422.7 nm. For this purpose use may also be made of a filter type flame photometer. The intensity of radiation either at the specified wavelength, or through the relevant filter is compared with that obtained for simulated standard solutions prepared from standard calcium carbonate solution.

NOTE — Water used in preparing standard solutions shall be double-distilled so that it produces no deflections of the galvanometer when the instrument is set for full scale deflection for 40 ppm of calcium concentration.

A-10.2.1 Apparatus

A-10.2.1.1 Flame photometer — Provided with arrangement for monochromatic light or a set of filters required for determination of calcium. The photometer comprises essentially a galvanometer, a photocell
or tube and a photo-multiplier tube. Other accessories are an atomizer-burner (or an atomizer and a burner) fed with town gas or liquefied petroleum gas (LPG) and compressed air (or oxygen, acetylene, etc.) in such a ratio that combustion of gas is complete.

A-10.2.1.2 Burette — of 25-ml nominal capacity and graduated in 0.05 ml of Class A accuracy (see IS : 1997-1982*).

A-10.2.1.3 Volumetric flasks — 1 000-ml, 250-ml and 100-ml nominal capacity, of Class A accuracy (see IS : 915-1975†).

A-10.2.2 Reagents

A-10.2.2.1 Concentrated hydrochloric acid — See IS : 265-1976‡.

A-10.2.2.2 Standard calcium solution — Weigh accurately 0.01 g of calcium carbonate and dissolve it by adding 10 ml of water and about 10 drops of concentrated hydrochloric acid. Make the volume to 100 ml with water. One millilitre of this solution is equivalent to 40 ppm of calcium.

A-10.2.2.3 Standard strontium nitrate solution — Weigh accurately 0.05 g of strontium nitrate. Dissolve it in water and make the volume to 100 ml with water.

A-10.2.2.4 Strontium nitrate sample solution — Weigh accurately 0.05 g of the sample of strontium nitrate, dissolve in water and make the volume to 100 ml with water.

A-10.2.3 Procedure — Adjust the flame photometer for analysis. Aspirate water through a capillary over the flame and using calcium filter adjust zero on the scale.

A-10.2.3.1 Aspirate standard calcium solution and by adjusting sensitivity, note the reading. Let it be \( a \).

A-10.2.3.2 Aspirate standard strontium nitrate solution and the sample solution in the same manner and note the readings. Let these be \( b \) and \( c \), respectively.

A-10.2.4 Calculation

\[
\text{Calcium [as Ca(NO}_3\text{)\text{2}], percent by mass} = \frac{(c - b) \times 40 \times 10^2 \times 10^2 \times 164}{a \times 10^6 \times 0.05 \times 40}
\]

Add to the above, known percentage of calcium nitrate in the standard strontium nitrate sample.

*Specification for burettes (second revision).
†Specification for one-mark volumetric flask (first revision).
‡Specification for hydrochloric acid (second revision).
A-11. DETERMINATION OF BARIUM

A-11.1 Outline of the Method — From the residue of barium and strontium nitrate left in A-10.1.2.1, barium is separated by precipitating as barium chromate from a faintly acidic solution, strontium remaining in solution.

A-11.2 Reagents

A-11.2.1 Ammonium Dichromate Solution — Dissolve 100 g of ammonium dichromate in 500 ml of water. Add 135 ml of 6 N ammonium hydroxide solution, filter, if necessary, and dilute to one litre.

A-11.2.2 Ammonium Acetate Solution — Dissolve 231 g of ammonium acetate in one litre of water or mix equal volumes of 6 N ammonium hydroxide and 6 N acetic acid leaving the solution slightly ammoniacal.

A-11.2.3 Dilute Acetic Acid — 6 N.

A-11.2.4 Dilute Nitric Acid — 2 N.

A-11.3 Procedure — Dissolve the residue of strontium nitrate and barium nitrate, obtained after the treatment with ether and alcohol in A-10.1.2.1, in water and make the solution up to 500 ml in a volumetric flask. Take 100 ml of this solution and dilute it to about 300 ml. Heat to boiling, add 10 drops of acetic acid and about 10 ml of ammonium dichromate solution (there should be an excess over theoretical amount necessary) and allow to stand for one hour. Decant off the supernatent liquid on Whatman filter paper No. 42 and wash with water containing 25 ml of ammonium dichromate solution per litre until the washings no longer give a precipitate with ammonium hydroxide and ammonium carbonate. Then wash with hot water containing 25 ml of ammonium acetate solution per litre until the last washing gives only a slight reddish-brown coloration with neutral silver nitrate solution.

A-11.3.1 The precipitate on the filter paper still contains a little strontium. Carefully wash it back into the vessel in which it was originally precipitated and dissolve any precipitate remaining on the filter paper in a little hot dilute nitric acid allowing it to run through the filter into the vessel containing the precipitate. Wash the filter with hot water till free from acid. Heat the precipitate till it dissolves in dilute nitric acid (about 6 ml is usually sufficient). Dilute the solution to 200 ml, heat to boiling, add 5 ml of ammonium acetate solution little by little and finally enough ammonium dichromate solution is added to cause the disappearance of the odour of acetic acid of the solution (usually about 10 ml are necessary). Allow the solution to stand for one hour. Cover the residue in the beaker with hot water. Allow to cool and filter through a tared sintered glass crucible No. G 4, and wash with cold water until the filtrate gives only slight opalescence with
neutral silver nitrate solution. Wash with dilute alcohol, dry at 110°C, cool and weigh as barium chromate. Repeat the operation of drying and cooling till constant mass is obtained.

A-11.3.2 Preserve the filtrate and washings for the estimation of strontium.

A-11.4 Calculation

Barium compounds, percent by mass = 100 \times 1.03 \times \frac{M_2 - M_1}{M}

where

- $M_2$ mass in g of the crucible with the precipitate,
- $M_1$ mass in g of the crucible, and
- $M$ mass in g of the material in the aliquot of the solution taken.

A-12. DETERMINATION OF SODIUM

A-12.1 Reagents

A-12.1.1 Concentrated Sulphuric Acid — See IS : 266-1977*.

A-12.1.2 Concentrated Hydrochloric Acid — See IS : 265-1976†.

A-12.1.3 Dilute Sulphuric Acid — 10 percent (v/v).

A-12.1.4 Absolute Alcohol — See IS : 321-1964‡.

A-12.2 Before the determination of sodium a qualitative test shall be carried out.

A-12.2.1 Qualitative Test for Sodium — Dissolve 2 g of the sample in 25 ml of water in a silica dish. Add 2 ml of concentrated sulphuric acid and slowly evaporate to near dryness. Take up with 20 ml of water. Filter through a washed filter paper Whatman No. 40. Evaporate the filtrate to dryness in the same dish and moisten with 2 drops of concentrated hydrochloric acid. Dip a clean platinum wire into the edge of a colourless Bunsen flame. When it is found to be colourless, remove from the flame and cool and dip it in the solution of the prepared sample. Again introduce it into the edge of the colourless Bunsen flame. The presence of sodium radical is established if the characteristic yellow colour is imparted to the flame. Estimate sodium compounds as prescribed in A-12.3.

* Specification for sulphuric acid (second revision).
† Specification for hydrochloric acid (second revision).
‡ Specification for absolute alcohol (revised).
A-12.3 Dissolve 5 g of the material in 200 ml of water, heat until boiling and add drop by drop 50 ml of dilute sulphuric acid. Continue boiling for about 20 minutes. Cool, add equal volume of absolute alcohol and allow to stand overnight. Then filter and wash the precipitate with alcohol-water mixture. Evaporate the filtrate and washings to dryness in a tared silica dish and ignite it at a dull red heat to remove sulphuric acid. Cool in a desiccator and weigh.

A-12.4 Calculation — Express the mass of the residue as sodium nitrate making correction for calcium as estimated in A-10 and record as a percentage of the original sample.

NOTE — Flame photometer may be used for determination of sodium where such facilities exist.

A-13. DETERMINATION OF AMMONIUM COMPOUNDS

A-13.0 Outline of the Method — Ammonium compounds are determined colorimetrically by comparing the colour produced by the material with Nessler's solution against that produced by a standard ammonium chloride solution.

A-13.1 Apparatus

A-13.1.1 Nessler Cylinders — 50 ml capacity.

A-13.2 Reagents

A-13.2.1 Potassium Iodide — crystals.

A-13.2.2 Mercuric Chloride Solution — saturated.

A-13.2.3 Potassium Hydroxide — solid.

A-13.2.4 Nessler's Solution — Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly with stirring, mercuric chloride solution until a slight permanent precipitate forms. Add 30 g of potassium hydroxide and when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

A-13.2.5 Sodium Hydroxide Solution — 5 N.

A-13.2.6 Standard Ammonium Chloride Solution — Dissolve 0.3141 g of ammonium chloride in water and make up to 1000 ml. One millilitre of this solution contains 0.1 mg of ammonia (as NH₃).

A-13.3 Procedure — Weigh accurately 1.0 g of the prepared sample and dissolve in 20 ml of water in a Nessler cylinder. Add 5 ml of 5 N sodium hydroxide solution and 1 ml of Nessler's solution. Make up the
solution to 50 ml. Carry out a control test in another Nessler cylinder with 1 ml of standard ammonium chloride solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture, and compare the colour.

A-13.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the test with the material is not deeper than that produced in the control test.

A-14. DETERMINATION OF STRONTIUM

A-14.1 Reagents

A-14.1.1 Dilute Nitric Acid — 5 N.

A-14.1.2 Dilute Ammonium Hydroxide — 5 percent (m/m).

A-14.1.3 Ammonium Carbonate Solution — 20 percent (m/v).

A-14.1.4 Dilute Hydrochloric Acid — 5 N.

A-14.1.5 Dilute Sulphuric Acid — 5 N.

A-14.2 Procedure — Collect the filtrate and washings obtained in A-11.2.2 and make up the volume to 500 ml in a measuring flask. Take an aliquot of it (50 ml equivalent to 0.1 g sample). Add 1 ml of dilute nitric acid. Make the solution slightly alkaline with the addition of dilute ammonium hydroxide and precipitate strontium as strontium carbonate by adding a slight excess of ammonium carbonate solution. Filter the precipitate in an asbestos ignited and tared Gooch crucible and wash with hot water. Dissolve the precipitate in dilute hydrochloric acid and reprecipitate strontium as strontium sulphate with the addition of dilute sulphuric acid. Add an equal volume of alcohol. Keep it overnight. Filter, wash with alcohol and dry the precipitate. Ignite and weigh as strontium sulphate till constant mass.

A-14.3 Calculation

\[
\text{Strontium nitrate, percent by mass} = 100 \times \frac{1.15 \times (M_2 - M_1)}{M}
\]

where

\( M_2 \) - mass in g of the crucible with the ignited precipitate,
\( M_1 \) = mass in g of the empty crucible, and
\( M \) = mass in g of the material in the aliquot of the solution taken.
APPENDIX B
(Clause 4.1)

SAMPLING OF STRONTIUM NITRATE FOR PYROTECHNIC COMPOSITIONS

B-1. GENERAL REQUIREMENTS

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be drawn in an exposed place.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments 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 thoroughly by suitable means.

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

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample container shall be sealed air-tight with a stopper after filling, and marked with all particulars of the material (sec 3.2) and the date of sampling.

B-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In single consignment of the material all the containers of the same type and size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different types and sizes of containers, the containers belonging to the same batch, type and size shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.
TABLE 2 SCALE OF SAMPLING
( Clause B-2.1.1 )

<table>
<thead>
<tr>
<th>LOT SIZE</th>
<th>SAMPLE SIZE</th>
</tr>
</thead>
<tbody>
<tr>
<td>$N$</td>
<td>$n$</td>
</tr>
<tr>
<td>(1)</td>
<td>(2)</td>
</tr>
<tr>
<td>Up to 15</td>
<td>3</td>
</tr>
<tr>
<td>16 to 25</td>
<td>4</td>
</tr>
<tr>
<td>26 to 50</td>
<td>5</td>
</tr>
<tr>
<td>51 to 100</td>
<td>7</td>
</tr>
<tr>
<td>101 and above</td>
<td>10</td>
</tr>
</tbody>
</table>

B-2.1.2 In order to ensure randomness of selection, random number tables (see IS : 4905-1968*) shall be used. In case such tables are not available, the following procedure is recommended for use:

Arrange the containers in the lot in a systematic order and starting from anyone, count them as 1, 2, 3, . . . etc, 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.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected, draw a small representative portion of the material not less than 100 g in mass.

B-3.2 Out of these portions, small but equal quantity of the material shall be taken and mixed thoroughly, to form a composite sample of mass not less than 150 g. The composite sample shall be divided into 3 equal parts, one for the purchaser, one for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into 3 equal parts and transferred to separate bottles, giving full identification particulars of the samples on the bottles. The material in each bottle constitutes an individual sample. One of these three sets (each containing one bottle representing each container sampled) shall be marked for the purchaser, another for the supplier and the third for the referee.

B-3.4 The referee test samples consisting of a composite sample and a test of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute between the two.

*Methods for random sampling.
B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of moisture, matter insoluble in water and total strontium nitrate shall be performed on each of the individual samples.

B-4.2 Tests for the determination of all the remaining characteristics given in 2 and Table 1 shall be carried out on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 From each set of the individual test results for moisture, matter insoluble in water and total strontium nitrate, the mean ($\bar{x}$) and range ($R$) of test results shall be computed separately (the range being defined as the difference between the maximum and the minimum values of the test results).

B-5.1.1 The lot shall be declared as conforming to the requirements of moisture and matter insoluble in water, if ($\bar{x} + 0.6 R$) as calculated from the test results is less than or equal to the relevant maximum value specified in Sl No. (i) and (ii) of Table 1.

B-5.1.2 The lot shall be declared as conforming to the requirements of total strontium nitrate if ($\bar{x} - 0.6 R$) as calculated from the relevant test results is greater than or equal to 97.0.

B-5.2 For declaring the conformity of the lot to the specification requirements of all other characteristics tested on the composite sample, the test results shall meet the corresponding requirements specified in Table 1.
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Printed at Printograph, New Delhi, India