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IS 12681 (1989): Sodium nitrate for explosives and pyrotechnics industry [CHD 26: Explosives and Pyrotechnics]



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*Indian Standard*

**SODIUM NITRATE FOR EXPLOSIVES AND  
PYROTECHNIC INDUSTRY — SPECIFICATION**

भारतीय मानक

विस्फोटक और आतिशबाजी उद्योग के लिए सोडियम नाइट्रेट — विशिष्ट

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**BUREAU OF INDIAN STANDARDS  
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NEW DELHI 110002**

## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 15 April 1989, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

Sodium nitrate has molecular formula  $\text{NaNO}_3$  and molecular weight 85. Sodium nitrate is one of the important raw materials in the manufacture of explosives and pyrotechnics. It is mainly used as an oxidizing agent in the manufacture of both NG based as well as slurry based explosives. Sodium nitrate is also used in glass manufacture and there is a separate standard for sodium nitrate for glass compositions ( IS 9157 : 1979 Specification for sodium nitrate and potassium nitrate for glass compositions ).

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 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## Indian Standard

# SODIUM NITRATE FOR EXPLOSIVES AND PYROTECHNIC INDUSTRY — SPECIFICATION

### 1 SCOPE

This standard prescribes requirements and methods of sampling and test for sodium nitrate for explosives and pyrotechnic industry.

### 2 REQUIREMENTS

#### 2.1 Description

The material shall be in the form of free-flowing fine colourless crystals, free from grit and other foreign matter.

2.1.1 The material shall have particle size as agreed to between the purchaser and the supplier.

2.2 The material shall also comply with the requirements given in Table 1 when tested according to methods given in Annex A. Reference to relevant clauses of Annex A is given in col 4 of Table 1.

### 3 PACKING AND MARKING

#### 3.1 Packing

The material shall be packed in clean, dry, air-tight packages as agreed to between the purchaser and the supplier.

#### 3.2 Marking

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

- a) Name of the material;
- b) Net mass of the material;
- c) Manufacturer's name and/or his trade-mark, if any; and
- d) Lot number and date of manufacture.

### 4 SAMPLING

4.1 The method of preparing representative samples of the material and the criteria for its conformity to this specification shall be as prescribed in Annex B.

**Table 1 Requirements for Sodium Nitrate for  
Explosives and Pyrotechnic Industry**  
( Clauses 2.2 and B-4.2 )

Sl No.	Characteristic	Requirement	Methods of Test ( Ref to Annex A )
(1)	(2)	(3)	(4)
1	Assay, percent by mass, <i>Min</i>	99.0	A-2
2	Moisture and volatile matter, percent by mass, <i>Max</i>	0.5	A-3
3	Matter insoluble in water, percent by mass, <i>Max</i>	0.03	A-4
4	Nitrites ( as $\text{NaNO}_2$ ), percent by mass, <i>Max</i>	0.05	A-5
5	Chlorides ( as $\text{NaCl}$ ), percent by mass, <i>Max</i> ,	0.1	A-6
6	Sulphates ( as $\text{Na}_2\text{SO}_4$ ), percent by mass, <i>Max</i>	0.25	A-7
7	Chlorates	Nil	A-8
8	Perchlorates ( as $\text{NaClO}_4$ ), percent by mass, <i>Max</i>	0.05	A-9
9	Iodates ( as $\text{NaIO}_3$ ), percent by mass, <i>Max</i>	0.02	A-10
10	Total calcium and magnesium or compounds of these metals calculated as oxides, percent by mass, <i>Max</i>	0.05	A-11
11	Ammonium compounds ( as $\text{NH}_4\text{NO}_3$ ), percent by mass, <i>Max</i>	0.05	A-12
12	pH of 10 percent solution	5.5—7.0	A-13

## ANNEX A

( Clause 2.2 )

## METHODS OF TEST FOR SODIUM NITRATE FOR EXPLOSIVES AND PYROTECHNIC INDUSTRY

## A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water 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 ASSAY

## A-2.0 General

Three methods, namely, ferrous ammonium sulphate method, ion-exchange method and ultraviolet spectrophotometric method have been given. However, in case of dispute UV spectrophotometric method shall be the referee method.

## A-2.1 Ferrous Ammonium Sulphate Method

## A-2.1.1 Reagents

A-2.1.1.1 *Ferrous ammonium sulphate*, 0.2 N.

A-2.1.1.2 *Potassium permanganate*, 0.1 N.

A-2.1.1.3 *Ammonium molybdate*, 10 percent aqueous solution.

A-2.1.1.4 *Sulphuric acid*, 28 N.

## A-2.1.2 Procedure

Weigh about 0.2 g of the material in a 500-ml flask and dissolve in water. Add 25 ml of 0.2 N ferrous ammonium sulphate solution and 5 ml of 10 percent aqueous solution of ammonium molybdate followed by gradual addition of 50 ml of 28 N sulphuric acid. Mix the contents of the flask and boil briskly for about 3-4 minutes. Cool the flask rapidly, dilute with water and again cool. Titrate the solution with 0.1 N potassium permanganate until the pink colour produced at the end point remains stable for about 5 minutes.

## A-2.1.3 Calculation

Calculate the excess of ferrous ammonium sulphate reacted with potassium permanganate (1 ml of 0.1 N  $\text{KMnO}_4 \equiv 0.005\ 585\ \text{g Fe}$ ). From this, find out the amount of ferrous ion converted to ferric ion by subtracting from total amount of ferrous salt taken (1 ml of 1N  $\text{FeSO}_4 \equiv 0.028\ 34\ \text{g of NaNO}_3$ ).

## A-2.2 Ultraviolet Spectrophotometric Method

## A-2.2.1 Reagents

A-2.2.1.1 *Demineralized water*

A-2.2.1.2 *Sodium nitrate GR*

## A-2.2.2 Equipment

A-2.2.2.1 *UV Spectrophotometer with 1-cm cells*

## A-2.2.3 Procedure

Weigh accurately about 1.5 g of the sample and transfer into a 100-ml volumetric flask. Dissolve by demineralized water and dilute to mark. Filter the solution if found hazy. Prepare a reference solution of sodium nitrate ( $\text{NaNO}_3$  GR) in the same manner. Take the absorbance measurement of both the solutions at 354 and 302 nm. Take absorbance readings of solvent (water) blank also.

## A-2.2.4 Calculation

Corrected absorbance of reference  $\text{NaNO}_3$  at 302 nm.

$$A_R = B - \frac{A}{2.57}$$

Corrected absorbance of sample at 302 nm.

$$A_S = D - \frac{C}{2.57}$$

$$\text{Percent sodium nitrate} = \frac{A_S \times 100}{A_R}$$

where

A = absorbance of reference sodium nitrate solution at 354 nm

B = absorbance of reference sodium nitrate solution at 302 nm,

C = absorbance of sample solution at 354 nm, and

D = absorbance of sample solution at 302 nm.

## A-2.3 Ion Exchange Method

## A-2.3.1 Principle

When a dilute solution of known quantity of sodium nitrate is passed through a suitable column containing strong cation exchanger in  $\text{H}^+$  form, the salt is converted to corresponding acid which is titrated against standard sodium hydroxide solution to determine the purity of sodium nitrate.

## A-2.3.2 Apparatus

The cation exchange column consists of a vertical glass tube about 2 cm bore, fitted at the lower

end with a stop-cock and containing a height of about 25 cm of cation exchange resin. The total length of the tube may be 60 cm or it may be widened into a funnel above the level of the resin. The resin is introduced into the column by mixing it with water and pouring the slurry into the tube so that no air bubbles are trapped between the particles. During use, the liquid level is not allowed to fall below the surface level of the resin.

### A-2.3.3 Preparation of the Column

The resin is washed by passing carbon dioxide free water down the column until 50 ml of elute requires not more than 0.05 ml of N/10 sodium hydroxide using phenolphthalein as the indicator.

**A-2.3.3.1** Up to four assays can normally be carried out by the column before regeneration. If a doubt exists as to the effectiveness of the column, the elute should be tested for the cation that should have been removed.

### A-2.3.4 Procedure

Dissolve 0.3 g of the material in 50 ml carbon dioxide free water and pass the solution through the cation exchange column at a rate of 3 to 4 ml per minute, collecting the elute in a titration flask. Wash the resin with carbon dioxide free water and titrate the elute with standard N/10 sodium hydroxide using bromothymol blue indicator as the washing proceeds until 50 ml of elute requires no further titration to the green end point.

### A-2.3.5 Calculation

$$\text{Percentage of sodium nitrate} = \frac{0.8499 \times v \times f}{m}$$

where

$v$  = volume in ml of standard sodium hydroxide required for the titration,

$f$  = factor of standard sodium hydroxide used, and

$m$  = mass of the sample taken for the test.

### A-2.3.6 Regeneration of Resin

The resin can be regenerated by leaving 4 N hydrochloric acid in the column overnight and passing a further 50 ml of hydrochloric acid through the column before the usual washing.

## A-3 DETERMINATION OF MOISTURE AND VOLATILE MATTER

### A-3.1 Procedure

Weigh accurately about 10 g of the material in a petri-dish. Keep the petri-dish in an air-oven maintained at  $110 \pm 2^\circ\text{C}$  for 2 hours. Cool the

petri-dish in a desiccator and weigh. Repeat the process of heating and cooling till constant mass is obtained.

### A-3.2 Calculation

$$\text{Moisture and volatile content, percent by mass} = \frac{(M_1 - M_2) 100}{M_1}$$

where

$M_1$  = mass in g of the material taken for the test, and

$M_2$  = mass in g of the material after drying.

## A-4 DETERMINATION OF MATTER INSOLUBLE IN WATER

### A-4.1 Procedure

Weigh accurately 20.00 g of the material and dissolve in about 250 ml of water. Filter through a tared sintered glass crucible (G. No. 4) and wash the residue thoroughly with water several times using small quantities of water to ensure removal of all soluble matter into the filtrate. Dry the residue at  $105 \pm 2^\circ\text{C}$  for 30 minutes, cool in a desiccator and weigh.

### A-4.2 Calculation

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

where

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

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

## A-5 DETERMINATION OF NITRITES

### A-5.1 Reagents

**A-5.1.1** *Potassium Permanganate*, 0.02N.

**A-5.1.2** *Sulphuric Acid*, 1 : 1 ( $v/v$ ).

### A-5.2 Procedure

Weigh accurately 25 g of the material, dissolve in water and dilute to 100 ml in a measuring flask. Pipette out 20 ml of 0.02 N potassium permanganate in 250 ml conical flask. Add 20 ml of water and 5 ml of 1 : 1 sulphuric acid. Heat to  $40^\circ\text{C}$  and titrate against sodium nitrite solution till the pink colour disappears.

### A-5.3 Calculation

Nitrite ( as sodium nitrite ), percent by mass

$$= \frac{0.0345 \times 10 \times N \times 100}{M \times V} \times 100$$



where.

$N$  = normality of potassium permanganate,

$M$  = mass in g of sodium nitrate taken for estimation,

$V$  = volume in ml of sodium nitrate used in titration.

## A-6 DETERMINATION OF CHLORIDES

### A-6.1 Reagents

**A-6.1.1 Standard Silver Nitrate Solution**, 0.1 N approx.

**A-6.1.2 Concentrated Nitric Acid**

**A-6.1.3 Ferric Ammonium Sulphate Indicator**, 10 percent (  $m/v$  ).

**A-6.1.4 Standard Ammonium Thiocyanate Solution**, 0.1 N approximately.

**A-6.1.5 Nitrobenzene**

### A-6.2 Procedure

Weigh accurately 2.0 g of the material and dissolve in about 50 ml of water. Add 10 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid and 5 ml of nitrobenzene. Shake vigorously for one minute. Add 2 ml of ferric ammonium sulphate indicator solution and titrate excess silver nitrate with standard ammonium thiocyanate solution shaking well between successive additions to the first persistent colour change.

### A-6.3 Calculation

Chlorides ( as Na Cl ), 
$$\text{percent by mass} = \frac{5.846 ( 10 N_1 - V N_2 )}{M}$$

where

$N_1$  = normality of standard silver nitrate solution,

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

$N_2$  = normality of standard ammonium thiocyanate solution, and

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

## A-7 DETERMINATION OF SULPHATES

### A-7.1 Reagents

**A-7.1.1 Concentrated Hydrochloric Acid**

**A-7.1.2 Barium Chloride Solution**, approximately 10 percent (  $m/v$  ).

### A-7.2 Procedure

Weigh accurately about 2.0 g of the sample and dissolve in 50 ml of water. Add 15 ml of concentrated hydrochloric acid and dilute to 200 ml with water. Filter, if necessary. Heat to boiling, add drop by drop, with constant stirring, 10 ml of barium chloride solution and allow to stand for about 2 hours. Filter through a tared sintered glass crucible ( G. No. 4 ), wash the precipitate thoroughly with hot water till free from chlorides and dry between 105 to 110°C to constant mass.

### A-7.3 Calculation

Sulphates ( as  $\text{Na}_2\text{SO}_4$  ), 
$$\text{percent by mass} = \frac{60.92 M_1}{M}$$

where

$M_1$  = mass in g of the barium sulphate precipitate, and

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

## A-8 DETERMINATION OF CHLORATES

### A-8.1 Reagents

**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 distilled water. Add 2 drops of saturated potassium chlorate solution, shake and allow to stand overnight. Filter off the blue sediment, store the reagent in a glass stoppered bottle in the dark.

### A-8.2 Procedure

Dissolve 5 g of the material in 40 ml of water, filter if necessary, and make up to 100 ml with aniline hydrochloride reagent.

**A-8.2.1** The material shall be considered to have passed the test if no blue colouration is developed in 10 minutes.

## A-9 DETERMINATION OF PERCHLORATES

### A-9.1 Apparatus

The apparatus consists of a wide-necked flask ( about 300 ml ) fitted with a cork carrying a reflux condenser, an inlet tube ( 6 mm bore ) reaching the bottom of the flask, and a short tube ( at least 12 mm bore ) fitted with a cork.

### A-9.2 Reagents

**A-9.2.1 Concentrated Hydrochloric Acid**

**A-9.2.2 Sulphur Dioxide**, gas.

**A-9.2.3 Dilute Sulphuric Acid**, approximately 12 N.

**A-9.2.4 Titanous Chloride Solution**, approximately 3 percent, prepared by diluting 10 ml of 15 percent (*m/v*) of titanous chloride solution to 50 ml with concentrated hydrochloric acid.

**A-9.2.5 Ammonium Thiocyanate Solution**, approximately 10 percent (*m/v*).

**A-9.2.6 Standard Ferric Ammonium Sulphate Solution**, approximately 0.1 N prepared by dissolving 48.22 g of ferric ammonium sulphate in 500 ml of freshly boiled water that has been acidified with 10 ml of dilute sulphuric acid (40 percent by mass). Make up the volume to 1 000 ml with freshly boiled water (Solution A).

a) Measure into a 200-ml conical flask 25 ml of freshly boiled water, 25 ml of 0.1 potassium bichromate solution and 15 ml of dilute sulphuric acid (40 percent by mass). Fit the conical flask with a rubber bung bored with two holes, through one of which passes a leading tube reaching, about 50 mm into the flask. Pass a rapid stream of carbon dioxide into the flask for several minutes in order to displace the air. Place the jet of the burette containing the titanous chloride solution in the second hole of the rubber bung, and whilst continuing to pass carbon dioxide, run 20 ml of the titanous chloride solution. Mix the liquid by swirling the flask gently and titrate the excess of titanous salt with Solution A added from a 25 ml burette, the jet of which is placed inside the second hole in the rubber bung. Towards the end of the titration, that is, when the dark colour due to the titanous salt has nearly disappeared, add 10 ml of ammonium thiocyanate solution and continue the addition of Solution A until a pink colour is obtained that is permanent for one minute. Maintain a slow stream of carbon dioxide throughout the titration.

b) In the same way, titrate 20 ml of titanous chloride solution with Solution A from a 50 ml burette but omitting 0.1 N potassium bichromate solution. From the volume of Solution A used in this titration, deduct the volume of ferric ammonium sulphate solution used in the first titration (this difference corresponds to 25 ml of 0.1 N potassium bichromate solution), and calculate the exact normality of ferric ammonium sulphate solution.

### A-9.3 Procedure

Weigh accurately about 5 g of the material and transfer it completely into the wide necked flask. Dissolve it in water and pass sulphur dioxide through the solution. Note the progress of the

reduction by the evolution of heat and the completion of reduction by the solution becoming cold (the time taken is generally 1½ to 2 hours). Remove the excess of sulphur dioxide completely with boiling and by the aid of a current of carbon dioxide passing through the liquid. Complete removal is indicated by the absence of odour of sulphur dioxide but as a precaution, continue the boiling gently for 30 minutes after the odour has disappeared. After cooling, add 20 ml of dilute sulphuric acid and dilute the solution with water to 150 ml.

Fit the flask to a reflux condenser and pass carbon dioxide through the liquid continuously until the end of the titration. Boil the solution for a few minutes to remove dissolved air. Add an exact volume (say, 10 ml) of titanous chloride solution by means of a pipette through the wide glass tube which is immediately closed with a small cork. Continue refluxing for one hour, after which remove the flask from the condenser and cool in water. Add 5 ml of ammonium thiocyanate solution, and then titrate with solution A through the glass-lined hole in the cork.

**A-9.3.1** Carry out a blank determination using potassium chlorate (perchlorate-free) and other reagents used in the test under A-9.3.

**A-9.3.2** Apparatus for titration in inert (carbon dioxide) atmosphere and for preservation and addition of titanous chloride solution are shown in Fig. 1 and 2, respectively.

### A-9.4 Calculation

$$\text{Perchlorate (as NaClO}_4\text{)} = \frac{1.53 (V_1 - V_2) N}{M}$$

where

$V_1$  = volume in ml of standard ferric ammonium sulphate solution required for the blank determination (see A-9.3.1),

$V_2$  = volume in ml of standard ferric ammonium sulphate solution (see A-9.3) required for test with the material,

$N$  = normality of standard ferric ammonium sulphate solution, and

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

## A-10 DETERMINATION OF IODATES

### A-10.1 Reagents

#### A-10.1.1 Standard Potassium Iodide Solution

Dissolve 0.65 g of potassium iodide in water and make up to one litre.

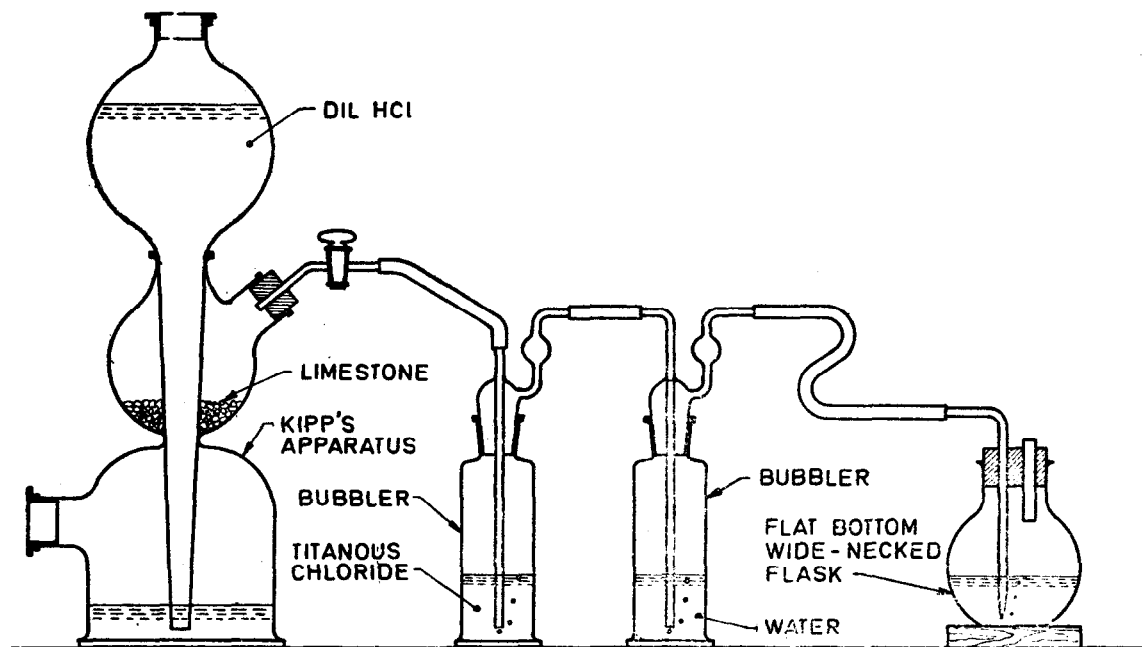


FIG. 1 APPARATUS FOR TITRATION IN AN INERT ( CARBON DIOXIDE ) ATMOSPHERE

**A-10.1.2** *Sodium Hydroxide*, one percent solution.

**A-10.1.3** *Hydrogen Peroxide*, 3 percent solution.

**A-10.1.4** *Sulphuric Acid*

**A-10.1.5** *Hydrogen Sulphide Gas*

**A-10.1.6** *Ortho-Toluidine Solution*, 1.0 g of ortho-toluidine dissolved in 150 ml of 95 percent alcohol.

#### **A-10.2 Procedure**

Take 10 g of sample separately in two beakers and dissolve in minimum quantity of water. Make it alkaline by using 1 percent sodium hydroxide. Add 10 ml of 3 percent hydrogen peroxide to oxidize nitrites. Reduce the bulk to about 20 ml by boiling. Neutralize by adding dilute sulphuric acid drop by drop using litmus paper as the indicator. In one beaker, pass hydrogen sulphide gas till it is saturated. Boil off excess of hydrogen sulphide and cool. To both beakers, add 0.5 ml of ortho-toluidine solution.

**A-10.2.1** Take the contents of the two beakers in two separate Nessler's cylinders and make the volume to 50 ml. Compare the turbidities with a series of standards using 1 to 10 ml of standard potassium iodide solution. The difference in turbidities between the two Nessler's cylinders is due to iodates and shall not exceed 2.5 ml of standard potassium iodide solution.

## **A-11 DETERMINATION OF CALCIUM AND MAGNESIUM**

### **A-11.0 General**

Calcium and magnesium may be determined by precipitating as the ferrocyanide in a 1 : 1 alcoholic mixture.

### **A-11.1 Reagents**

**A-11.1.1** *Ammonium Ferrocyanide*, one percent solution.

**A-11.1.2** *Ethyl Alcohol*

**A-11.1.3** *Standard Calcium Carbonate Solution*

Dissolve 1.778 g of calcium carbonate in 50 ml of 1 N hydrochloric acid and dilute to 1 litre with water. 1.0 ml of this solution is equivalent to 1 mg of calcium oxide.

### **A-11.2 Procedure**

Take 10 g of sample and dissolve in 40 ml of water, add 50 ml of ethyl alcohol, 5 ml of one percent ammonium ferrocyanide and stir. Take it in a Nessler's cylinder and make up the volume.

**A-11.2.1** Compare the turbidity with a series of standards using 1 to 5 ml of standard calcium carbonate solution. Turbidity of sample solution shall not be more than that of the standard using 5 ml standard solution.

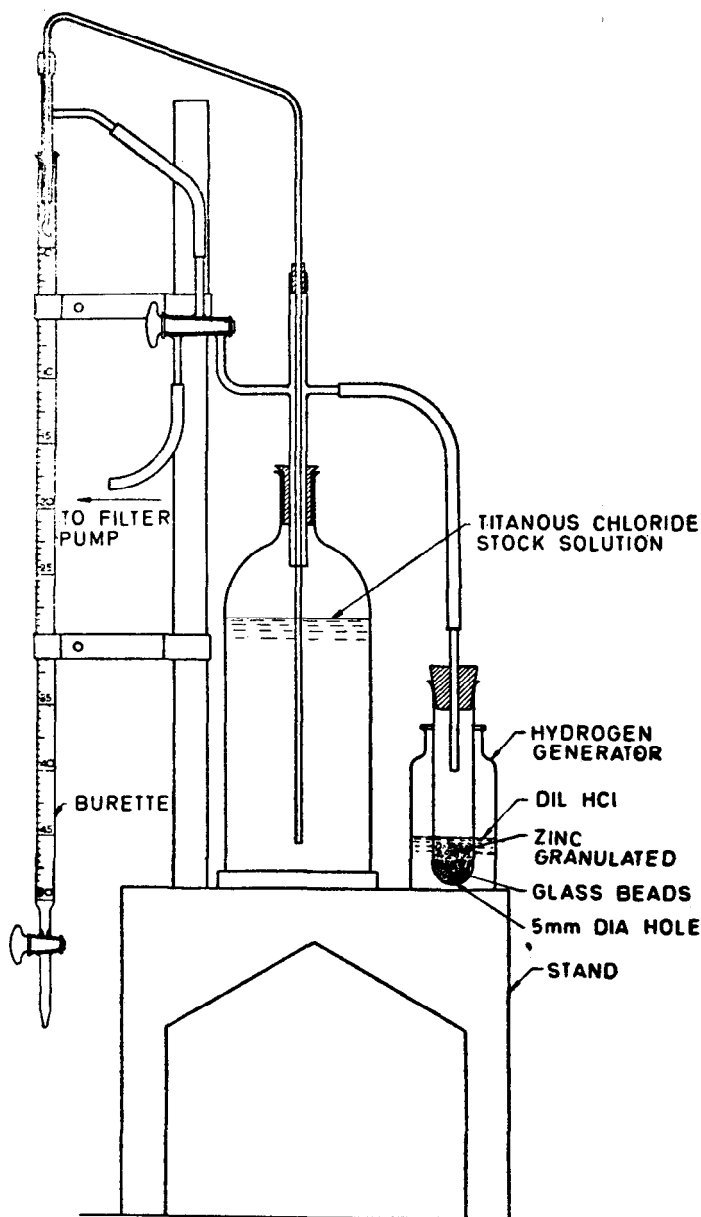


FIG. 2 APPARATUS FOR PRESERVATION AND ADDITION OF TITANOUS CHLORIDE SOLUTION

## A-12 DETERMINATION OF AMMONIUM COMPOUNDS

### A-12.1 Reagents

#### A-12.1.1 Nessler's Solution

Dissolve 100 g of mercuric iodide ( $\text{Hg I}_2$ ) and 70 g of potassium iodide (KI) in a small quantity of water. Add this mixture slowly, with stirring, to a cool solution of 160 g sodium hydroxide dissolved in 500 ml of water. Dilute to one litre. Store in rubber stoppered borosilicate glass-ware and away from sunlight.

A-12.1.2 Ammonium Chloride Solution, 0.15 g of ammonium chloride dissolved in one litre of water.

### A-12.2 Procedure

Dissolve 5 g of sample in water and make up the volume to 100 ml in a Nessler cylinder. Add 2 ml of Nessler's solution and after 3 minutes, compare the colour developed with that of other Nessler cylinders containing known volumes of the standard ammonium chloride solution treated in a similar manner.

1 ml NH<sub>4</sub> Cl           ≡ 0.001 percent NH<sub>3</sub>  
( 0.15 g per litre )       ( on 5 g sample )

**A-12.3 Calculation**

Ammonium compounds  
( as NH<sub>4</sub>NO<sub>3</sub> ),  
percent by mass           = ( 0.001 percent  
                                  NH<sub>3</sub> × 4.71 )

**A-13 DETERMINATION OF pH****A-13.1 Procedure**

Dissolve 10.0 g of the material in 100 ml of water and measure the pH of the solution by means of a suitable pH meter using glass electrodes.

**ANNEX B**

( Clause 4.1 )

**SAMPLING OF SODIUM NITRATE FOR EXPLOSIVES AND PYROTECHNICS INDUSTRY****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 ( see 2.2 ) 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 a 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 sizes 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**

Lot Size N	Sample Size n
(1)	(2)
Up to 15	3
16 „ 25	4
26 „ 50	5
51 „ 100	7
101 and above	10

**B-2.1.2** In order to ensure randomness of selection, random number tables 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 any one, 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. The referee sample 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.

#### **B-4 NUMBER OF TESTS**

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

**B-4.2** Tests for determination of all the remain-

ing characteristics given in col 2 of Table 1 shall be carried out on the composite sample.

#### **B-5 CRITERIA FOR CONFORMITY**

##### **B-5.1 For Individual Samples**

For declaring the conformity of the lot:

- a)  $\bar{X} + 0.6 R$  shall be less than or equal to the maximum specified requirements, and
- b)  $\bar{X} - 0.6 R$  shall be greater than or equal to the minimum specified requirements.

where

$\bar{X}$  = mean value of test results, and

$R$  = range of results.

##### **B-5.2 For Composite Sample**

For declaring the conformity of the lot to the requirements of all characteristics tested on the composite sample, the test results shall satisfy the corresponding specified requirements.

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