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Draft Indian Standard

SODIUM NITRATE AND POTASSIUM NITRATE FOR GLASS

COMPOSITIONS - SPECIFICATION

(First Revision)

भारतीय मानक मसौदा

कांच के संयोजन के लिए सोडियम नाइट्रेट और पोटेशियम नाइट्रेट - विशिष्ट

(पहला पुनरीक्षण)

ICS 81.040.30

Glass, Glassware & Laboratoryware Sectional Committee, CHD 10

Last date for Comments: 20 Dec 2022

FOREWORD

(Formal clauses shall be added later)

Sodium nitrate and potassium nitrate are used in batch compositions of glass, enamel frits and glazes in which their chief function is to oxidize organic matter which may contaminate batch materials, prevent reduction of some of the batch constituents, help maintain colours and accelerate the melting and fining processes.

This standard was originally published in 1979. The first revision has been undertaken to bring it in line with the technological changes that may have happened over the last four decades. Several editorial changes such as the inclusion of the ICS No., Hindi Title, Reference Clause, etc. have also been incorporated.

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.

SODIUM NITRATE AND POTASSIUM NITRATE FOR GLASS

COMPOSITIONS - SPECIFICATION

(*First Revision*)

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for sodium nitrate and potassium nitrate for glass compositions.

2 REFERENCES

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

<i>IS No.</i>	<i>Title</i>
IS 2 : 2022	Rules for Rounding Off Numerical Values (<i>Second Revision</i>)
IS 1260 (Part 1) : 1973	Pictorial Marking for Handling and Labelling of Goods: Part 1 Dangerous Goods (<i>First Revision</i>)
IS 1070 : 1992	Reagent grade water - Specification (<i>Third Revision</i>)
IS 264 :2005	Nitric acid - Specification (<i>Third Revision</i>)
IS 265 :2021	Hydrochloric Acid — Specification (<i>Fifth Revision</i>)
IS 1997 : 2008/ ISO 385 : 2005	Laboratory glassware - Burettes (<i>Third Revision</i>)
IS 915 : 2012/ ISO 1042: 1998	Laboratory glassware - One - Mark volumetric flasks (<i>Third Revision</i>)
IS 4905 : 2015/ ISO 24153: 2009	Random sampling and randomization procedures (<i>First Revision</i>)

3 GRADES

3.1 The material shall be of two grades as follows:

Grade 1 – suitable for optical and special glasses, and

Grade 2 – suitable for commercial glasses.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of white crystalline powder, free from grit, visible impurities and foreign matter.

4.2 The material, dried in accordance with the method prescribed in **A-2.1**, shall comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in **Annex A**. Reference to relevant clauses of **Annex A** is given in col 7 of the table.

5 PACKING AND MARKING

5.1 Packing

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

5.2 Marking

The packages shall be marked with the following:

- a) Name, grade and net mass of the material;
- b) Name of the manufacturer or his recognized trade-mark, if any; and
- c) Lot number and date of manufacture.
- d) In addition, the packages shall be labelled with the symbol indicating the danger of oxidizing substances [*see* IS 1260 (Part 1)] and the words ‘ HANDLE WITH CARE ‘.

5.2.1 BIS Certification Marking

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

6 SAMPLING

6.1 The 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 and potassium nitrate for glass compositions (Clause 4.2)						
Sl. No.	Characteristic	Requirement for				Methods of test, Ref to CL No. of Annex A
		Sodium Nitrate		Potassium Nitrate		
		Grade 1	Grade 2	Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)	(6)	(7)

i.	Moisture and volatile matter, percent by mass, <i>Max</i>	0.25	1.0	0.25	1.0	A-2
		← [on dry basis, see 4.2] →				
ii.	Matter insoluble in water, percent by mass, <i>Max</i>	0.05	0.25	0.05	0.25	A-3
iii.	Carbonates	To pass the test		To pass the test		A-4
iv.	Nitrites	To pass the test		To pass the test		A-5
v.	Chlorides (as Cl), percent by mass, <i>Max</i>	0.2	0.5	0.2	0.5	A-6
vi.	Sulphate (as SO ₄), percent by mass*, <i>Max</i>	0.1	0.2	0.1	0.2	A-7
vii.	Copper*	To pass the test		To pass the test		A-8
viii.	Iron (as Fe), percent by mass, <i>Max</i>	0.01	0.05	0.01	0.05	A-9
ix.	Manganese*	To pass the test		To pass the test		A-10
x.	Potassium compounds (as K), percent by mass, <i>Max</i>	0.5	1.0	–	–	A-11
xi.	Sodium compounds (as Na), percent by mass, <i>Max</i>	–	–	0.5	1.0	A-11
xii.	Sodium Nitrates (as NaNO ₃), percent by mass, <i>Min</i>	99.2	98.0	–	–	A-12
xiii.	Potassium nitrates (as KNO ₃), percent by mass, <i>Min</i>	–	–	99.2	98.0	A-12

- **The Committee may consider deleting these requirements as there is No source of these elements in manufacturing process.**

ANNEX A
(Clause 4.2, 6.1)
**ANALYSIS OF SODIUM NITRATE AND POTASSIUM NITRATE
FOR GLASS COMPOSITIONS**

A-1 QUALITY OF REAGENTS

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

NOTE – ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF MOISTURE AND VOLATILE CONTENT

A-2.1 Procedure – Crush about 50 g of the material to pass 500-micron IS Sieve. Weigh accurately in a tared petri dish and spread in a layer of uniform thickness. Keep the dish in an air-oven, maintained at $140\pm 2^{\circ}\text{C}$, for 3 hours, cool in a desiccator, and weigh. Repeat the process of heating and cooling till constant mass is reached. Preserve the dried material for further tests.

A-2.2 Calculation

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

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-3 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure – Weigh accurately 20.00 g of the material, reserved in A-2.1, 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. Dry the residue at $140\pm 2^{\circ}\text{C}$ for 30 minutes, cool in a desiccator and weigh.

A-3.1.1 Cool the filtrate, transfer to a 500-ml volumetric flask, dilute with water to the mark and reserve for further tests.

A-3.2 Calculation

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

Where,

M_1 = mass in g of the residue, and

M = mass in g of the dried material (A-2.1) taken for the test.

A-4 TEST FOR CARBONATES

A-4.1 Reagents

A-4.1.1 *Dilute Hydrochloric Acid* - 10 percent (v/v).

A-4.1.2 *Lime Water* - Freshly prepared and filtered saturated solution of lime in water.

A-4.2 Procedure - Transfer about 2 g of the dried material (A-2.1) into a test tube, add 20 ml of dilute hydrochloric acid and allow the gas to pass through clear lime water.

A-4.2.1 The material shall be taken as not having satisfied the requirement of the test if there is effervescence with evolution of a colourless gas which turns clear lime water turbid.

A-5 TEST FOR NITRITES

A-5.1 Reagents

A-5.1.1 *Potassium Iodide Solution* - 5 percent (m/v).

A-5.1.2 *Carbon Tetrachloride*

A-5.1.3 *Acetic Acid* – glacial.

A-5.2 Procedure – Pipette 25 ml of the solution of the dried material, prepared in A-3.1.1, into a test tube, add 2 to 3 drops of potassium iodide solution and 1 ml each of carbon tetrachloride and glacial acetic acid. Shake for 5 minutes. Simultaneously, carry out a blank test in another test tube using 25 ml of water and the same quantities of the reagents as with the material.

A-5.2.1 The material shall be taken as having satisfied the requirement of the test if the intensity of the violet colour produced in the organic layer in the test is not greater than that produced in the blank test.

A-6. DETERMINATION OF CHLORIDES

A-6.1 Reagents

A-6.1.1 *Standard Silver Nitrate Solution* – 0.1 N approximately.

A-6.1.2 *Concentrated Nitric Acid* – (see IS 264)

A-6.1.3 *Ferric Ammonium Sulphate Indicator Solution* – 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 – Pipette 50 ml of the solution of the dried material, prepared in **A-3.1.1**, into a 250-ml conical flask. Add 10 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid and 5 ml of nitrobenzene. Shake vigorously for 1 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

$$\text{Chlorides (as Cl), percent by mass} = \frac{3.546(10N_1 - VN_2)}{M}$$

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 dried material in the aliquot taken for the test.

A-7 DETERMINATION OF SULPHATES

A-7.1 Reagents

A-7.1.1 Concentrated Hydrochloric Acid – (see IS : 265)

A-7.1.2 Dilute Hydrochloric Acid – approximately 10 percent (v/v).

A-7.1.3 Barium Chloride Solution – approximately 10 percent (m/v).

A-7.2 Procedure – Pipette 50 ml of the solution of the dried material, prepared in **A-3.1.1**, into a 400-ml beaker, 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. tared sintered glass crucible (G. No. 4), Filter through a 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

$$\text{Sulphates (as SO}_4\text{), percent by mass} = \frac{41.6M_1}{M}$$

Where,

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

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

A-8 TEST FOR COPPER

A-8.1 Reagents

A-8.1.1 Citric Acid Solution – Dissolve 25 g of citric acid in 100 ml of water.

A-8.1.2 Ethylene Diamine Tetra Acetate (Disodium Salt) Solution – 4 percent (*m/v*) in water.

A-8.1.3 Cresol Red Indicator.

A-8.1.4 Dilute Ammonium Hydroxide – 50 percent (*v/v*).

A-8.1.5 Sodium Diethyl Dithiocarbamate Solution – Dissolve 0.1 g of sodium diethyl dithiocarbamate in 100 ml of water.

A-8.1.6 Carbon Tetrachloride – copper free.

A-8.2 Procedure – Pipette 25 ml of the solution of the dried material, prepared in **A-3.1.1**, into a 100-ml volumetric flask and add 5 ml of citric acid solution. Add 5 ml of EDTA disodium salt solution, render slightly alkaline with dilute ammonium hydroxide (*pH* about 8.5) using cresol red indicator and cool to room temperature. Add 5 ml of sodium diethyl dithiocarbamate solution followed by 2 ml, of carbon tetrachloride and shake for 10 seconds. Simultaneously carry out a blank test in another 100-ml volumetric flask, using 25 ml of water and the same quantities of reagents as with the material.

A-8.2.1 The material shall be taken as having satisfied the requirement of the test if the intensity of the yellow colour produced with the material is not greater than that produced in the blank test.

A-9 TEST FOR IRON

A-9.1 Apparatus

A-9.1.1 Spectrophotometer

A-9.2 Reagents

A-9.2.1 Buffer Solution – Dissolve 270 g of anhydrous sodium acetate in 500 ml of water, add 240 ml of 17 M glacial acetic acid and dilute to 1000 ml with water.

A-9.2.2 Hydroxylamine Hydrochloride Solution – Dissolve 10 g in water and dilute to 100 ml. This solution should be freshly prepared.

A-9.2.3 o-Phenanthroline Solution – Dissolve 0.5 g of o-phenanthroline in 100 ml of water and store in a dark bottle with a ground-glass stopper.

A-9.2.4 Standard Iron Solution – Dissolve 7.02 g of ammonium ferrous sulphate $[(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}]$ in water containing 10 ml of concentrated sulphuric acid and dilute with water to 1000 ml. Dilute 10 ml of this solution to 100 ml in a volumetric flask. One millilitre of this diluted solution is equivalent to 0.1 mg of iron (as Fe).

A-9.3 Procedure

A-9.3.1 Transfer 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 ml respectively of the standard iron solution, prepared in **A-9.2.4**, to 50-ml volumetric flasks. Add 2 ml of hydroxylamine hydrochloride solution, 1 ml of o-phenanthroline solution and adequate volume of buffer solution to bring the pH value to about 5. Dilute the various solutions to the mark with water. After 30 minutes, determine the absorbance against a reagent blank at 510 nm using a 1-cm cell. Draw calibration curve between absorbance and concentration of iron (in mg/ml).

A-9.3.2 Pipette 25 ml of the solution of the dried material, prepared in **A-3.1.1**, into a 50-ml volumetric flask, add 2 ml of hydroxylamine hydrochloride solution and proceed as in **A-9.3.1** determining the absorbance after 30 minutes against a reagent blank at 510 nm using a 1-cm cell, Read the concentration of iron (in mg/ml) of the solution from the curve (A-9.3.1) and calculate the total amount of iron (as Fe).

NOTE – *If quantities of iron involved are high, measurement of absorbance may be carried out using aliquots of more dilute solutions.*

A-10 TEST FOR MANGANESE

A-10.1 Reagents

A-10.1.1 *Dilute Sulphuric Acid* – 50 percent (v/v).

A-10.1.2 *Syrups Phosphoric Acid* – manganese free.

A-10.1.3 *Potassium Periodate* – powder.

A-10.2 Procedure – Pipette 25 ml of the solution of the dried material, prepared in **A-3.1.1**, into a 100-ml beaker. Add 5 ml of dilute sulphuric acid followed by 5 ml of syrupy phosphoric acid. Heat to boiling, add 0.5 g of potassium periodate and continue boiling for another minute. Maintain the solution just below boiling point for about 10 minutes and observe the colour of the solution. Simultaneously, carry out a blank test in another beaker using 25 of water and the same quantities of reagents as with the material.

A-10.2.1 The material shall be taken as having satisfied the requirement of the test if the intensity of violet colour produced with the material is not greater than that produced in the blank test.

A-11 DETERMINATION OF POTASSIUM CONTENT IN SODIUM NITRATE AND SODIUM CONTENT IN POTASSIUM NITRATE

A-11.0 General – Potassium or sodium content is determined with the help of a flame emission spectrophotometer by measuring the intensity of radiation emitted at 767 nm by potassium or at 589 nm by sodium. For this purpose use may also be made of a filter type flame photometer. In both the cases, however, 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 potassium chloride and sodium chloride solutions.

NOTE – *Water used in preparing standard solutions shall be double-distilled so that it produces no deflection of the galvanometer when the instrument is set for full scale deflection for 10 ppm of potassium or 5 ppm of sodium concentration.*

A-11.1 Apparatus

A-11.1.1 Flame Photometer – Provided with arrangement for monochromatic light or a set of filters required for determination of potassium and sodium. The photometer comprises essentially a galvanometer, a photo cell or tube and a photo-multiplier tube. Other accessories are an atomizer-burner (or an atomizer and a burner) fed with fuel gas or liquified petroleum gas (LPG) and compressed air (or oxygen, acetylene, etc.) in such a ratio that combustion of gas is complete.

A-11.1.2 Burette – of 25 ml nominal capacity and graduated in 0.05 ml, of Class A accuracy (see IS 1997).

A-11.1.3 Volumetric Flasks – two of 1000 ml, two of 250 ml and 14 of 100 ml nominal capacity, of Class A accuracy (*see* IS 915).

A-1 1.2 Reagents

A-11.2.1 Hydrochloric Acid - 1: 1 (v/v).

A-11.2.2 Standard Sodium Solutions – Weigh accurately 0.5084 g of sodium chloride (NaCl), previously dried to constant mass at $110 \pm 2^\circ\text{C}$. Dissolve in water, transfer to a 1000-ml volumetric flask, dilute to the mark with water and mix.

A-11.2.2.1 Transfer 10 ml of this solution to a 100-ml volumetric flask, dilute up to the mark with water and mix. This diluted solution contains 20 ppm of sodium (as Na).

A-11.2.2.2 Transfer 2.5, 2.0, 1.5, 1.0 and 0.5 ml respectively of the standard sodium solution prepared in **A-11.2.2.1** to five 100-ml volumetric flasks. Add 37.5 ml each of potassium solution prepared in **A-11.2.3** to the five flasks, dilute up to the mark with water and mix. These solutions contain 5, 4, 3, 2 and 1 ppm respectively of sodium (as Na).

A-11.2.3 Standard Potassium Solutions – Weigh accurately 0.3814 g of potassium chloride (KCl), previously dried at $110 \pm 2^\circ\text{C}$ to constant mass. Dissolve in water, transfer to a 1000-ml volumetric flask, dilute up to the mark with water and mix.

A-11.2.3.1 Transfer 10 ml of this solution to a 100-ml volumetric flask, dilute up to the mark with water and mix. This diluted solution contains 20 ppm of potassium (as K).

A-11.2.3.2 Transfer 5.0, 4.0, 3.0, 2.0, 1.5, 1.0 and 0.5 ml respectively of the standard potassium solution: prepared in **A-11.2.3.1** to seven 100-ml volumetric flasks. Add 26.0 ml each of the

sodium solution prepared in **A-11.2.2**, to the seven flasks, dilute up to the mark with water and mix. These solutions contain 10, 8, 6, 4, 3, 2 and 1 ppm respectively of potassium (as K).

A-11.3 *Setting-up the Apparatus* – Switch the instrument on and wait for its stabilization. Adjust the slit and wavelengths at 767 nm for potassium or 589 nm for sodium or place the filters in position, as the case may be.

A-11.3.1 Pour water in the spray cup, open its tap and turn the compressed air or oxygen on. Adjust the tap and pressure to establish atomization of water in a fine spray. Open the gas tap and light the burner. Adjust gas, if necessary, to get complete combustion. Next switch the photocell on and adjust the indicator needle or the indicator light spot, as the case may be, to bring it to the 'zero' position.

A-11.3.2 Atomize 10 ppm potassium (**A-11.2.3.2**) or 5 ppm sodium (**A-11.2.2.2**) solutions in place of water (**A-11.3.1**) and adjust the deflection to full scale. Atomize water once again to check the indicator needle or light spot rests at 'zero'. If it shifts from 'zero' again adjust it to 'zero'. Thus when the instrument reads 'zero' with water and shows full scale deflection for 10 ppm potassium (as K) or 5 ppm sodium (as Na) solution, it is ready for use.

NOTES

1 – Atomization of any solution should always be followed by atomization of double-distilled water till the indicator comes to zero position.

2 – Do not disturb the setting of the atomizer or burner so that rates of flow are constant for a series of determinations.

A-11.4 Procedure

A-11.4.1 *Preparation of the Sample Solution* – Weigh accurately 0.5 g of the sample previously dried at 110 f 2°C to constant mass, Dissolve in water, transfer to a 250-ml volumetric flask, dilute up to the mark with water and mix.

A-11.4.2 *Calibration Graph and Determination* – Without disturbing the setting of the instrument, atomize standard potassium (**A-11.2.3.2**) or sodium (**A-11.2.2.2**) solutions of different concentrations in an ascending order and plot the readings of the indicator against concentrations and draw the graph. Next atomize the sample solution (**A-11.4.1**) and note the deflection and calculate concentration of potassium or sodium as the case may be as illustrated in the following example:

Example:

Suppose reading for 10 ppm K solution is 90.0, that for 8 ppm K solution is 72.0: and that for sample solution is T (somewhere between 96.0 and 72.0)

Similarly, suppose reading for 4 ppm Na solution is 80.0, that for 5 ppm Na solution is 100.0; and that for sample solution is T (somewhere between 80.0 and 100.0)

Then concentration of potassium (C_K) in the sample solution is expressed as:

$$C_k = 8 + \frac{10-8}{90-72} \times (T - 72) \text{ ppm}, \text{ or}$$

$$C_k = 10 - \frac{10-8}{90-72} \times (90 - T) \text{ ppm}$$

Similarly, concentration of sodium (C_{Na}) in the sample solution is expressed as:

$$C_{Na} = 4 + \frac{(5-4)}{100-80} \times (T - 80) \text{ ppm}, \text{ or}$$

$$C_{Na} = 5 - \frac{5-4}{100-80} \times (100 - T) \text{ ppm}$$

A-12 DETERMINATION OF SODIUM NITRATE AND POTASSIUM NITRATE

A-12.1 Reagents

A-12.1.1 Devarda's Alloy – Powder of alloy of copper 50 parts, aluminium 45 parts and zinc 5 parts by mass.

A-12.1.2 Standard Sulphuric Acid – approximately 0.2 N.

A-12.1.3 Sodium Hydroxide Solution –20 percent (m/v).

A-12.1.4 Standard Sodium Hydroxide Solution – approximately 0.2 N.

A-12.1.5 Methyl Orange Indicator Solution – Dissolve 0.05 g of methyl orange in 100 ml of water.

A-12.2 Procedure – Pipette 25 ml of the solution of the dried material, prepared in **A-3.1.1**, into a 500-ml round bottom boiling flask (A), add 200 ml of water, and 3 g of Devarda's alloy powder and fit up the apparatus as illustrated in Fig. 1, filling 100 ml of standard sulphuric acid in the receiver. Introduce 10 ml of sodium hydroxide solution (**A-12.1.3**) into the round bottom flask, through the thistle funnel (B) immediately closing its tap (F), Warm gently to start the reaction and allow to stand for 1 hour, so that evolution of gas has ceased and the reduction of nitrate to ammonia is complete. Then boil the contents of the flask gently and continue distillation till only 40 to 50 ml of liquid remains in the boiling flask. Open the tap (F) of the thistle funnel before removing the flame. Rinse the condenser tube (D) collecting the washings in the receiver (E). Finally, titrate the contents of the receiver against standard sodium hydroxide solution (**A-12.1.4**) using methyl orange as indicator.

A-12.3 Calculation

a) Sodium nitrate (as NaNO_3), percent by mass =
$$\frac{8.499(100N_1 - VN_2)}{M}$$

b) Potassium nitrate (as KNO_3), percent by mass =
$$\frac{10.111(100N_1 - VN_2)}{M}$$

where,

N_1 = normality of standard sulphuric acid,

V = volume in ml of the standard sodium hydroxide solution consumed in titration,

N_2 = normality of the standard sodium hydroxide solution, and

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

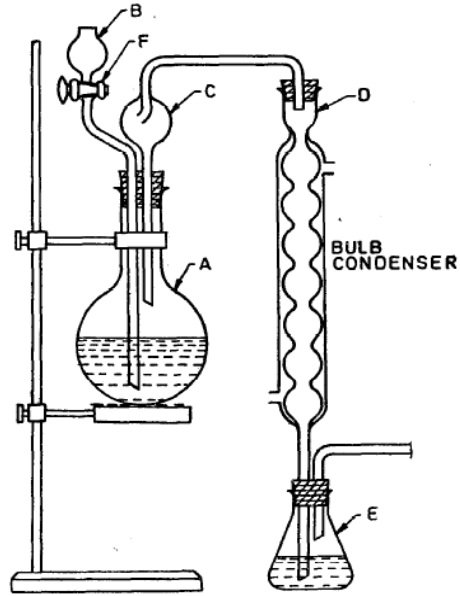


FIG. 1 APPARATUS ASSEMBLY FOR DETERMINATION OF SODIUM AND POTASSIUM NITRATE

ANNEXURE B

(Clause 6.1)

**SAMPLING OF SODIUM NITRATE AND POTASSIUM NITRATE FOR GLASS
COMPOSITIONS**

B-1 SCALE OF SAMPLING

B-1.1 Lot – In a single consignment, all packages of the same size and containing material of the same grade and belonging to the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of packages, the packages belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-1.2 For ascertaining conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately.

B-1.3 The number of packages to be selected from a lot shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

(Clause B-1.3)

Number of package in the <i>LOT</i>	Sampling Sizes
N	n
(1)	(2)
Up to 15	3
16 to 50	4
51 to 100	5
101 to 150	7
150 and above	10

B-1.3.1 The packages shall be selected from the lot at random. In order to ensure randomness of selection, reference may be made to IS 4905:1960*. In case this standard is not available the following procedure may be adopted:

Starting from any package 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 . Every rth package thus counted shall be withdrawn till the required number of packages is obtained.

B-2 PREPARATION OF TEST SAMPLES

B-2.1 From each of the packages selected draw a small representative portion of the material. The quantity of material so drawn shall be sufficient to make triplicate determinations for all the characteristics given in the specification.

B-2.2 Out of these portions, a small but approximately equal quantity of the material shall be taken and mixed thoroughly to form a composite sample. The composite sample shall be divided into 3 equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-2.3 The remaining portion of the material from each package 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 shall constitute an individual test sample. One each of these three sets (each set containing one bottle representing each package sampled) shall be marked for the purchaser, the supplier and the referee respectively.

B-2.4 The referee test sample consisting of a composite test sample and a set of individual test 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 the case of a dispute between the two.

B-3 NUMBER OF TESTS

B-3.1 Tests for the determination of sodium nitrate content, potassium nitrate content, copper, iron and manganese shall be conducted on each of the individual test samples.

B-3.2 Tests for the determination of all the remaining characteristics given in the specification shall be carried out on the composite test sample.

B-4 CRITERIA FOR CONFORMITY

B-4.1 From the test results on individual samples the average (\bar{X}) and the range (\bar{R}) of the test results shall be computed (range R is defined as the difference between the maximum and minimum values of the test results).

B-4.1.1 The lot shall be declared as conforming to the requirements tested on individual samples if the following conditions are satisfied:

<i>Characteristic</i>	<i>Criteria for Conformity</i>
i. Sodium nitrate content	$\bar{x} - 0.6R$ is equal to or greater than 99.2 for Grade 1 $\bar{x} - 0.6R$ is equal to or greater than 98.0 for Grade 2
ii. Potassium nitrate content	$\bar{x} - 0.6R$ is equal to or greater than 99.2 for Grade 1 $\bar{x} - 0.6R$ is equal to or greater than 98.0 for Grade 2
iii. Copper	All individual test samples shall pass the test given in A-8.
iv. Iron	All individual test samples shall pass the test given in A-9.
v. Manganese	All individual test samples shall pass the test given in A-10.

B-4.2 For declaring the conformity of the lot to the requirements of the remaining characteristics, the test results on the composite test sample shall meet the corresponding specified requirements.