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Indian Standard
SPECIFICATION FOR
POTASSIUM NITRATE FOR EXPLOSIVE AND
PYROTECHNIC COMPOSITIONS

(Second Revision)

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AMENDMENT NO. 1 SEPTEMBER 2003
TO
IS 301 : 1982 SPECIFICATION FOR POTASSIUM
NITRATE FOR EXPLOSIVE AND PYROTECHNIC
COMPOSITIONS
(*Second Revision*)

(*Page 8, clause B-5.2*) – Substitute the following for the existing:

'B-5.2 Procedure — Weigh about 100 g of prepared sample and dissolve in adequate quantity of water. Heat, if necessary, cool and allow to settle. Decant the supernatant liquid carefully and dry the residue. Add 30 ml aqua regia, heat to boiling and boil for 10 min. Allow to cool and dilute with water. Filter through a tared filter paper, wash the residue with water free from chloride and dry at 105 to 110°C to constant mass.'

(CHD 26)

Indian Standard
SPECIFICATION FOR
POTASSIUM NITRATE FOR EXPLOSIVE AND
PYROTECHNIC COMPOSITIONS
(Second Revision)

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Indian Standard
**SPECIFICATION FOR
POTASSIUM NITRATE FOR EXPLOSIVE AND
PYROTECHNIC COMPOSITIONS**
(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 29 January 1982, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 This Indian Standard was originally issued in 1951 and revised in 1963. In the first revision, the limits for potassium nitrate content, water insoluble organic matter and matter insoluble in aqua regia had been included. A flame photometer method for the determination of sodium was prescribed as an alternate method. In the present revision new methods for the determination of chlorides, chlorates, perchlorates and potassium nitrate contents have been included.

0.3 This standard contains clauses **3.3** and **4.1** which call for agreement between the purchaser and the supplier.

0.4 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*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for potassium nitrate used in the explosive and pyrotechnic compositions.

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

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2. GRADES

2.1 The material shall be of two grades, namely, Grade 1 and Grade 2.

3. REQUIREMENTS

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

3.2 Moisture and Volatile Content – When tested according to the method prescribed in Appendix A, the moisture and volatile content of the material shall be as follows:

- a) For Grade 1, percent by mass, *Max* 0.05
- b) For Grade 2, percent by mass, *Max* 0.20

3.2.1 Subject to agreement between the purchaser and the supplier, moisture and volatile content in excess of specified limit may be allowed, provided the material satisfies the other requirements of this standard.

3.3 Particle Size – The material shall comply with the requirements for sieve analysis as agreed to between the purchaser and the supplier.

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

4. PACKING AND MARKING

4.1 Packing – The material shall be packed in sound, clean and dry packages which shall ensure safe transportation and complete freedom from accidental contamination. The type and size of packages shall be subject to agreement between the purchaser and the supplier.

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

- a) Name and grade of the material;
- b) Tare and net mass;
- c) Name of the manufacturer and/or his recognized trade-mark, if any;
- d) Date of manufacture; and
- e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

TABLE 1 REQUIREMENTS FOR POTASSIUM NITRATE FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS

(Clause 3.4)

SL No.	CHARACTERISTIC	REQUIREMENT (ON DRY BASIS)		METHOD OF TEST
		Grade 1	Grade 2	(REF TO CL No. IN APPENDIX B)
(1)	(2)	(3)	(4)	(5)
i)	Hygroscopicity, percent by mass, <i>Max</i>	0.25	1.00	2
ii)	Matter insoluble in water, percent by mass, <i>Max</i>	0.05	0.1	3
iii)	Water insoluble organic matter, percent by mass, <i>Max</i>	0.02	0.02	4
iv)	Matter insoluble in aqua regia, percent by mass, <i>Max</i>	0.02	0.02	5
v)	Grit	Nil	Nil	5
vi)	Metallic radicals other than potassium and sodium	To pass test	To pass test	6
vii)	Acidity	do	do	7
viii)	Alkalinity	do	do	8
ix)	Chlorides (as KCl), percent by mass, <i>Max</i>	0.02	0.15	9
x)	Chlorates	To pass test	To pass test	10
xi)	Perchlorates (as $KClO_4$), percent by mass, <i>Max</i>	0.1	0.1	11
xii)	Sulphates (as K_2SO_4), percent by mass, <i>Max</i>	0.10	0.10	12
xiii)	Ammonium compounds (as NH_3)	To pass test	0.05 percent by mass, <i>Max</i>	13
xiv)	Sodium compounds (as $NaNO_3$)	do	0.10	do14
xv)	Nitrites (as KNO_2)	do	0.10	do15
xvi)	Potassium nitrate (as KNO_3), percent by mass, <i>Min</i>	99.50	98.75	16

4.2.1 The packages shall also be labelled with the symbol indicating the danger of oxidizing substances specified in IS : 1260 (Part I)-1973*.

4.2.2 The product may also be marked with Standard Mark.

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

5. SAMPLING

5.1 **Preparation of Test Samples** – The method of drawing representative test samples of the material, number of tests to be performed and the criteria of finding out the conformity of the material to the requirements of the specification shall be as prescribed in Appendix C.

APPENDIX A

(Clauses 3.2 and 3.4)

DETERMINATION OF MOISTURE AND VOLATILE CONTENT

A-1. PROCEDURE

A-1.1 Weigh accurately 10 to 15 g of the material, crushed as prescribed in B-1 into a tared weighing dish. Keep the dish in an oven maintained at 140±2°C for 2 hours. Cool in a desiccator and weigh till constant mass is obtained.

A-1.2 Calculation

$$\text{Moisture and volatile content, percent by mass} = 100 \times \frac{M_1 - M}{M_1}$$

where

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

*Pictorial marking for handling and labelling of goods: Part I Dangerous goods (first revision).

APPENDIX B

(Clause 3.4)

METHODS OF TEST FOR POTASSIUM NITRATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

B-0. QUALITY OF REAGENTS

B-0.1 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.

B 1. PREPARATION OF THE SAMPLE

B-1.1 Procedure — Crush 150 g of the material to pass 1.00-mm IS Sieve. Spread this crushed material in a petri dish so that the layer of the material is not more than one centimeter in thickness and dry as presented in **A-1.1**. Cool and preserve this prepared sample in a desiccator for subsequent tests.

B-2. DETERMINATION OF HYGROSCOPICITY

B-2.1 Procedure — Weigh accurately about 5 g of the prepared sample in a petri dish of approximately 6 cm diameter, spread evenly and expose it to an atmosphere of 90 percent relative humidity at $27 \pm 1^\circ\text{C}$ (*see* Note) for 24 hours. Weigh again.

NOTE — The test atmosphere of 90 percent relative humidity at $27 \pm 1^\circ\text{C}$ may be obtained as follows:

Place in a desiccator an aqueous sludge of hydrated zinc sulphate or sodium chloride so that the area of the surface of the sludge is not less than 75 percent of the floor area of the desiccator. Supports for keeping the sample in the desiccator shall be so arranged that there is minimum obstruction for the diffusion of water vapours within the desiccator and the height of the exposed sample above the zinc sulphate sludge does not exceed 10 cm. The desiccator shall be maintained at a temperature of $27 \pm 1^\circ\text{C}$.

B-2.2 Calculation

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

where

M_2 = mass in g of the material after exposure, and

M_1 = mass in g of the *prepared sample* taken for the test.

*Specification for water for general laboratory use (*second revision*).

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B-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

B-3.1 Procedure — Weigh accurately about 10 g of the *prepared sample*. Dissolve by stirring in 25 ml of water. Filter through a tared filter paper; wash the residue thoroughly to free it from all soluble matter and dry at 105 to 110°C to constant mass. Preserve the residue for the test in **B-4**.

B-3.2 Calculation

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

where

M_1 = mass in g of the dried residue, and

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

B-4. DETERMINATION OF WATER INSOLUBLE ORGANIC MATTER

B-4.1 Procedure — Transfer the residue preserved in **B-3.1** to a tared crucible and ignite till constant mass is obtained.

B-4.2 Calculation

$$\text{Water insoluble organic matter, percent by mass} = 100 \times \frac{M_1 - M_2}{M_3}$$

where

M_1 = mass in g of the dried residue, obtained in **B-3.1**;

M_2 = mass in g of the ignited residue; and

M_3 = mass in g of the *prepared sample* taken for the test in **B-3.1**.

B-5. DETERMINATION OF GRIT

B-5.1 Reagents

B-5.1.1 Aqua Regia – Prepared by mixing 3 volumes of concentrated hydrochloric acid with one volume of concentrated nitric acid.

B-5.2 Procedure — Weigh accurately about 10 g of the *prepared sample* and dissolve by stirring in 30 ml of aqua regia. Heat to boiling and dilute with water to 100 ml. Filter through a tared filter paper, wash the residue thoroughly with water till free from chlorides and dry at 105 to 110°C to constant mass.

B-5.2.1 Transfer the residue on the filter paper to a 63-micron IS Sieve with a light camel hair brush. Examine if any residue is retained on it and carry out the soda glass scratching test as prescribed in **B-5.3**.

B-5.3 Soda Glass Scratching Test – Take a portion of the residue, retained on 63-micron IS Sieve, in between two clean glass slides and press the slides by hand along the length and breadth of the glass. Examine whether any scratch on the glass is obtained. If there is a scratch on the glass, then the residue is termed as grit.

B-5.4 Calculation

$$\begin{array}{l} \text{Matter insoluble in aqua regia,} \\ \text{percent by mass} \end{array} = 100 \frac{M_1}{M_2}$$

where

M_1 = mass in g of the dried residue, and

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

B-5.4.1 Report whether any residue is retained on the 63-micron IS Sieve.

B-6. TEST FOR METALLIC RADICALS OTHER THAN POTASSIUM AND SODIUM

B-6.1 Reagents

B-6.1.1 Ammonium Hydroxide — 1 : 4 (v/v).

B-6.1.2 Hydrogen Sulphide Gas

B-6.1.3 Sodium Hydroxide Solution - 5 percent (m/v).

B-6.1.4 Titan Yellow – Dissolve 0.15 g of titan yellow in 400 ml cold water. Filter off any insoluble matter and dilute to 500 ml.

B-6.1.5 Ammonium Oxalate Solution – 10 percent (m/v).

B-6.1.6 Ammonium Carbonate Solution — 10 percent (m/v).

B-6.2 Procedure — Dissolve about 5 g of the *prepared sample* in about 20 ml of water. Test the solution obtained as in **B-6.2.1**, **B-6.2.2**, and **B-6.2.3**.

B-6.2.1 To about 5 ml of the solution, add about 2 ml of ammonium hydroxide and pass in a stream of hydrogen sulphide gas.

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B-6.2.2 To about 5 ml of the solution, add about 3 ml of sodium hydroxide solution and about 3 ml of titan yellow solution. Mix well.

B-6.2.3 To about 5 ml of the solution, add about 2 ml of ammonium hydroxide and 2 ml each of ammonium oxalate and ammonium carbonate solutions. Warm and allow to stand for a few minutes.

B-6.3 The material shall be taken to have passed the test if:

- a) no precipitate is obtained in **B-6.2.1**,
- b) no red lake or red colour is obtained in **B-6.2.2**, and
- c) no precipitate is obtained in **B-6.2.3**.

B-7. TEST FOR ACIDITY

B-7.1 Reagents

B-7.1.1 Methyl Orange Indicator Solution— Dissolve 0.01 g of methyl orange in 100 ml of water.

B-7.1.2 Phenolphthalein Indicator Solution – Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit conforming to IS : 323-1959*, and dilute to 100 ml with water.

B-7.1.3 Standard Sodium Hydroxide Solution – 0.01 N.

B-7.2 Prepared Solution – In a volumetric flask dissolve 10.0 g of the *prepared sample* in a sufficient volume of carbon dioxide-free water to produce 100 ml of the solution.

B-7.3 Procedure

B-7.3.1 For Grade 1

- a) *Acidity to methyl orange*—To 10 ml of the *prepared solution* add 3 drops of methyl orange indicator solution. The material shall be considered as having passed the test if no red coloration is developed.
- b) *Acidity to phenolphthalein* – Titrate 10 ml of the *prepared solution* against standard sodium hydroxide solution using 3 to 5 drops of phenolphthalein indicator solution. The material shall be considered to have passed the test if not more than 1.0 ml of standard sodium hydroxide solution is required for the titration.

*Specification for rectified spirit (*revised*).

B-7.3.2 For Grade 2

- a) *Acidity of methyl orange* — Titrate 20 ml of the *prepared solution* against standard sodium hydroxide solution using 3 to 5 drops of methyl orange indicator solution. The material shall be considered to have passed the test if not more than 0.4 ml of standard sodium hydroxide solution is required for the titration.
- b) *Acidity to phenolphthalein* — Titrate 10 ml of the *prepared solution* against standard sodium hydroxide solution using 3 to 5 drops of phenolphthalein indicator solution. The material shall be considered to have passed the test if not more than 2 ml of standard hydroxide solution is required for the titration.

B-8. TEST FOR ALKALINITY

B-8.1 Reagents

B-8.1.1 Phenolphthalein Indicator Solution — Same as in **B-7.1.2**.

B-8.2 Procedure — To 10 ml of the *prepared solution* as obtained in **B-7.2**, add 3 drops of phenolphthalein indicator solution. The material shall be considered as having passed the test if no red coloration is developed.

B-9. DETERMINATION OF CHLORIDES

B-9.0 Two methods are prescribed, namely, volumetric method and automatic chloride titrator method. In case of dispute volumetric method shall be the referee method.

B-9.1 Volumetric Method

B-9.1.1 Reagents

B-9.1.1.1 Standard silver nitrate solution — 0.1 N.

B-9.1.1.2 Concentrated nitric acid – see IS : 264-1976*.

B-9.1.1.3 Ferric alum indicator solution — saturated.

B-9.1.1.4 Standard ammonium thiocyanate solution — 0.1 N.

B-9.1.1.5 Nitrobenzene

B-9.1.2 Procedure — Weigh accurately about 5 g of the *prepared sample* and dissolve in about 80 ml of water. Filter the residue, if any, through a folded filter paper and wash thoroughly with water, collecting the filtrate and washings in 250-ml conical flask. Add with a pipette 25 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid and 10 ml of nitrobenzene. Shake vigorously and add 2 ml of ferric alum indicator solution. Titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change.

*Specification for nitric acid (*second revision*).

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B-9.1.3 Calculation

$$\text{Chlorides (as KCl), percent by mass} = \frac{0.7446 (25 - V)}{M}$$

where

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

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

B-9.2 Automatic Chloride Titrator Method

B-9.2.0 Principle – The concentration of chloride ions is quantitatively determined by electrometric titration with silver ions generated in solution coulometrically. The end point of the titration is determined amperometrically.

A constant direct current is passed between a pair of silver generator electrodes. Electrochemical oxidation at the anode releases silver ions at a steady rate into the titration solution. When all the chloride has combined with silver ions, the presence of free silver ions causes an abrupt increase in current between a pair of silver indicator electrodes.

B-9.2.1 Reagents

B-9.2.1.1 Nitric acid-acetic acid reagent – To about 80 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.

B-9.2.1.2 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.

B-9.2.1.3 Standard potassium chloride solution — Weigh accurately about 0.39 g of dry potassium chloride, dissolve in water and dilute to 100 ml.

B-9.2.2 Procedure — Clean the electrodes of the instrument and rinse with water thoroughly. Weigh accurately 0.1 to 0.5 g of the sample directly in the titration vessel. Add 4 ml of nitric acid-acetic acid reagent and 0.2 ml gelatin reagent.

B-9.2.2.1 Prepare standard by taking 0.1 ml of standard potassium chloride solution and 4 ml of nitric acid-acetic acid reagent and 0.2 ml of gelatin reagent. Also prepare blank solution.

*Specification for nitric acid (*second revision*).

†Specification for acetic acid (*second revision*).

B-9.2.2.2 Treat the sample solution, standard solution as prepared in **B-9.2.2.1** and the blank according to the instructions of the instrument and record the time.

B-9.2.3 Calculation

$$\text{Chloride (as KCl), percent by mass} = \frac{(B - C) \times M_1 \times 100}{(A - C) \times M}$$

where

B = time in seconds for potassium nitrate sample solution,

C = time in seconds for blank,

*M*₁ = mass in g of potassium chloride present in 0.1 ml of standard potassium chloride solution,

A = time in seconds for standard solution, and

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

B-10. TEST FOR CHLORATES

B-10.1 Reagent

B-10.1.1 Aniline Hydrochloride Solution – Dissolve 18 g of distilled 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, store the reagent in a glass stoppered bottle in the dark.

B-10.2 Procedure — Dissolve 5 g of the prepared sample in 50 ml of warm water, filter, if necessary, and make up to 100 ml with aniline hydrochloride reagent.

B-10.2.1 The material shall be considered to have passed the test if no blue colouration is developed in 10 minutes.

B-11. DETERMINATION OF PERCHLORATES

B-11.1 Reagents

B-11.1.1 Ammonium Chloride — solid.

B-11.1.2 Dilute Nitric Acid

B-11.1.3 Standard Silver Nitrate Solution — 0.02 N.

B-11.1.4 Ferric Alum Indicator Solution — saturated.

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B-11.1.5 *Standard Ammonium Thiocyanate Solution* — 0.02 N.

B-11.1.6 *Nitrobenzene*

B-11.2 Procedure — Weigh accurately 10 g of *prepared sample* and transfer to a hard glass test tube fitted with a rubber cork carrying a glass tube. Heat the test tube with a burner till oxygen begins to evolve. Continue heating for about 15 minutes. Disconnect the glass tube and allow to cool. Dissolve the contents in a small quantity of water and transfer to a conical flask. Add about 100 ml of water and acidify the solution with 2 ml of dilute nitric acid. Add 10 ml of silver nitrate solution and 5 to 10 ml of nitrobenzene to form a layer. Add 2 ml of ferric alum indicator. Titrate the solution with ammonium thiocyanate solution until brown colour appears. From the volume of ammonium thiocyanate solution consumed, Calculate the total chlorides as a percentage of the mass of the sample taken.

B-11.3 Calculation — Calculate potassium perchlorate content on the basis that 1 ml of 0.02 N ammonium thiocyanate is equivalent to 0.002 772 g of potassium perchlorate.

B-12. DETERMINATION OF SULPHATES

B-12.1 Reagents

B-12.1.1 *Concentrated Hydrochloric Acid* — see IS : 265-1976*.

B-12.1.2 *Dilute Hydrochloric Acid* – approximately 10 percent (m/v).

B-12.1.3 *Barium Chloride Solution* — approximately 10 percent (m/v).

B-12.2 Procedure – Weigh accurately about 5 g of the *prepared sample* and dissolve in 50 ml of water. Add 15 ml of concentrated hydrochloric acid and evaporate the solution to dryness. Moisten the residue with 10 ml of dilute hydrochloric acid, dilute to 200 ml with water and filter, if necessary. Heat the solution to boiling and add drop by drop 10 ml of barium chloride solution. Boil the solution for 15 minutes and then allow to stand for 4 hours. Filter through a tared Gooch crucible. Wash the precipitate thoroughly with hot water till free from chlorides and dry to constant mass at 105 to 110°C.

B-12.3 Calculation

$$\text{Sulphates (as } K_2SO_4 \text{), percent by mass} = 71.65 \times \frac{M_1}{M}$$

where

M_1 = mass in g of the precipitate obtained, and

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

*Specification for hydrochloric acid (*second revision*).

B-13. TEST FOR AMMONIUM COMPOUNDS

B-13.1 For Grade 1

B-13.1.1 Reagent

B-13.1.1.1 Sodium hydroxide solution — approximately 5 N.

B-13.1.2 Procedure — Moisten a few grams of the *prepared sample* on a clock-glass with sodium hydroxide solution and cover the clock-glass with another one, to the under side of which adheres a piece of moistened red litmus paper.

B-13.1.2.1 The material shall be taken to have passed the test if the litmus paper does not turn blue.

B-13.2 For Grade 2

B-13.2.1 Apparatus

B-13.2.1.1 Nessler cylinders — 50-ml capacity.

B-13.2.2 Reagents

B-13.2.2.1 Sodium hydroxide solution – approximately 5 N.

B-13.2.2.2 Nessler solution – Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly with stirring saturated 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.

B-13.2.2.3 Standard ammonium chloride solution – Dissolve 0.314 1 g of ammonium chloride in water and make up to 1 000 ml. One millilitre of this solution is equivalent to 0.1 mg of ammonia (as NH_3).

B-13.2.3 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 sodium hydroxide solution and one millilitre of Nessler solution. Make up the solution to 50 ml. Carry out a control test in another Nessler cylinder with 5 ml of standard ammonium chloride solution in place of the material and the same quantities of other reagents. Compare the colour produced in the two cylinders.

B-13.2.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

B-14. DETERMINATION OF SODIUM COMPOUNDS

B-14.1 For Grade 1

B-14.1.1 Reagents

B-14.1.1.1 Concentrated hydrochloric acid— see IS : 265-1976*.

B-14.1.2 Procedure — Dissolve 1.0 g of the *prepared sample* in 5 ml of water and acidify with concentrated hydrochloric acid. Dip a clean platinum wire into the solution of the material. Introduce the platinum wire into the edge of a non-luminous Bunsen flame.

B-14.1.2.1 The material shall be taken to have passed the test if the yellow flame characteristic of sodium is not obtained.

B-14.2 For Grade 2

B-14.2.0 General— Two methods, namely, Method A and Method B are prescribed. In Method A, sodium content is estimated by any suitable flame photometer which, when operated according to the manufacturer's instructions, will determine the sodium content of potassium nitrate to an accuracy of one percent absolute. Method B is the gravimetric method which, in case of dispute, shall be taken as the referee method.

B-14.2.1 Method A

B-14.2.1.1 Apparatus

Flame photometer – Set up and operate in accordance with the maker's instructions.

B-14.2.1.2 Reagents

- a) *Standard sodium solution*— Dissolve 1.271 0 g of sodium chloride (previously dried at 250 to 350°C to constant mass) in water and make up the solution to 1 000 ml.
- b) *Standard potassium nitrate solution* — Dissolve 100.0 g of twice crystallized potassium nitrate (previously dried at 100 to 105°C to constant mass) in water and make up the solution to 1 000 ml.

*Specification for hydrochloric acid (*second revision*).

B-14.2.1.3 Preparation of test solution — Measure by means of a pipette the following volumes of standard sodium solution and standard potassium nitrate solution into 250-ml volumetric flasks. Dilute the content of each flask to the mark and mix well.

Test Solution	Volume in ml of Standard	Volume in ml of Standard
	Sodium Solution	Potassium Nitrate Solution
Blank	Nil	50
A	2.5	50
B	5.0	50
C	7.5	50
D	10.0	50

B-14.2.1.4 Procedure — Set the instrument by spraying the blank solution and adjusting the galvanometer to zero and then spraying the test solution D and adjusting the galvanometer to read 100. Take readings for test solutions A, B and C and prepare a graph of sodium content against deflection. Determine the sodium content of the prepared sample by measuring the deflection of a 2 percent solution, setting the instrument with solution D and the blank solution before each determination.

B-14.2.2 Method B

B-14.2.2.1 Reagents

- a) *Hydrogen chloride gas*
- b) *Magnesium uranyl acetate solution* — In a 250-ml conical flask dissolve 9.0 g of uranyl acetate [$\text{UO}_2 (\text{COOCH}_3)_2 \cdot 2\text{H}_2\text{O}$] and 6 ml of glacial acetic acid in sufficient water to make up the volume to 100 ml. Warm to about 70°C and stir until solution is complete. In another flask dissolve 60.0 g of magnesium acetate [$\text{Mg} (\text{COOCH}_3)_2 \cdot 2\text{H}_2\text{O}$] and 6 ml of glacial acetic acid in sufficient water to produce 100 ml of the solution, warm to 70°C and stir until solution is complete. Mix both the solutions and cool to 20°C. Allow the solution to stand at this temperature for at least 2 hours and then filter through a dry filter paper into an amber coloured bottle. Protect the solution from direct sunlight and filter before use.
- c) *Wash alcohol* — Saturate rectified spirit (see IS : 323-1959*) with dry sodium magnesium uranyl acetate and filter.

*Specification for rectified spirit (revised).

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B-14.2.2.2 Procedure — Weigh accurately about 5 g of the *prepared sample* in a flask and dissolve in 15 ml of water. Evaporate till the solution is just saturated and does not deposit any crystals on cooling in ice water. Keep the flask containing the cold solution in ice water and pass hydrogen chloride gas into it through a tube sufficiently wide to remain unblocked. When the solution is saturated, stir to break up any lumps and move the crystals from the side of the flask and the tube into the bottom of the flask. Filter using gentle suction. Wash the residue on the filter funnel several times with small portions of the ice-cold filtrate. Suck the filter dry after each addition of the filtrate, concentrate this filtrate taking care that no crystals separate. Depending on the quantity of sodium present, add rapidly to the filtrate 50 to 60 ml of magnesium uranyl acetate solution. Immerse the flask in a bath maintained at $20 \pm 1^\circ\text{C}$ and stir vigorously for 30 to 45 minutes. Filter the precipitate immediately through a tared sintered glass crucible (G No. 4) using gentle suction, and after all the solution has run through, wash the precipitate 4 to 5 times with successive 5 ml portions of wash alcohol. Dry the crucible with its contents in an oven maintained at 105 to 110°C for 30 minutes and weigh as sodium magnesium uranyl acetate.

B-14.2.2.3 Calculation

$$\begin{array}{l} \text{Sodium compounds (as NaNO}_3 \text{),} \\ \text{percent by mass} \end{array} = 5.66 \times \frac{M_1}{M}$$

where

M_1 = mass in g of the dried precipitate, and

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

B-15. TEST FOR NITRITES

B-15.1 For Grade 1

B-15.1.1 Reagents

- a) *Potassium iodide solution* – 10 percent (*m/v*).
- b) *Chloroform*
- c) *Glacial acetic acid* – see IS : 695-1975*.

B-15.1.2 Procedure — Dissolve 1.0 g of the *prepared sample* in 10 ml of water, add 2 drops of potassium iodide solution and one millilitre each of chloroform and glacial acetic acid. Shake gently for 5 minutes.

*Specification for acetic acid (*second revision*).

B-15.1.2.1 The material shall be taken to have passed the test if no violet colour develops in the chloroform layer.

B-15.2 For Grade 2

B-15.2.1 Apparatus

B-15.2.1.1 *Nessler cylinders* — 50-ml capacity.

B-15.2.2 Reagents

- a) *Concentrated hydrochloric acid* — see IS : 265-1976*.
- b) *Standard potassium nitrite solution* — Dissolve exactly 1.0 g of potassium nitrite in water, and make up to 1 000 ml. One millilitre of the solution is equivalent to 0.001 g of nitrites (as KNO_2).
- c) *Dimethylaniline hydrochloride solution* — Dissolve 10 ml of dimethylaniline in 100 ml of 1 : 1 dilute hydrochloric acid.

B-15.2.3 Procedure — Dissolve 5.0 g of the prepared sample in 25 ml of water and filter, if necessary. Wash the filter paper and transfer both the filtrate and washings into a Nessler cylinder. Add 4 drops of concentrated hydrochloric acid and 0.5 ml of dimethylaniline hydrochloride solution and make up the volume to 50 ml. Carry out a control test in another Nessler cylinder using 5 ml of standard potassium nitrite solution in place of the material and the same quantities of other reagents; compare the colour after 20 minutes.

B-15.2.3.1 The limit prescribed shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

B-16. DETERMINATION OF POTASSIUM NITRATE

B-16.0 General — Two methods have been prescribed, namely, Lunge nitrometer method and Kjeldahl method.

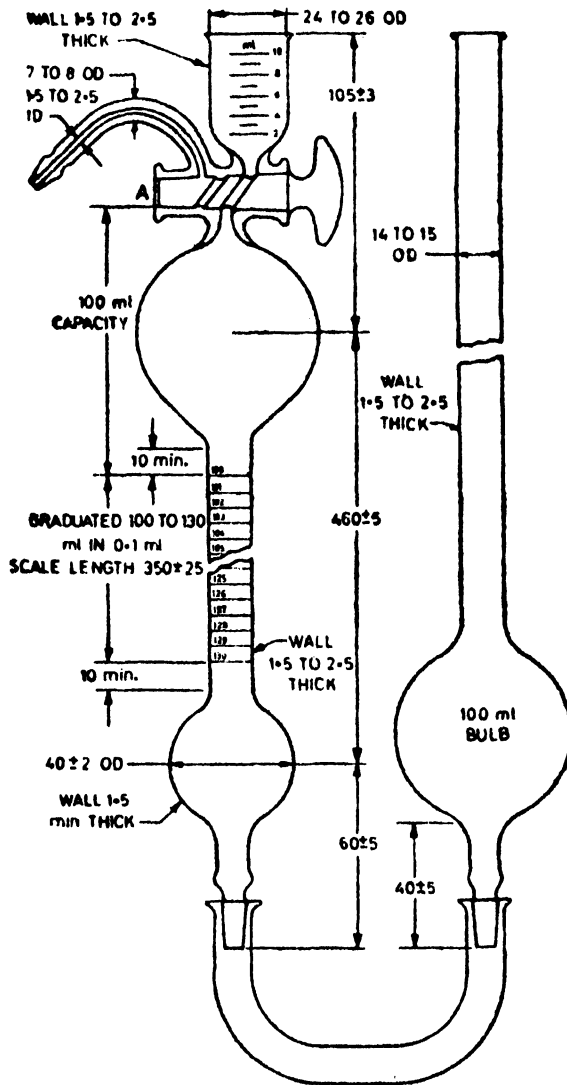
B-16.1 Lunge Nitrometer Method

B-16.1 Apparatus

B-16.1.1.1 *Lunge nitrometer* — Consisting of a burette, fitted at the top with a three-way tap A and a cup-shaped funnel, and communicating at the bottom by means of a rubber tubing with a levelling tube. Details of construction are shown in Fig. 1.

*Specification for hydrochloric acid (*second revision*).

IS : 301 - 1982



All dimensions in millimetres.

FIG. 1 LUNGE NITROMETER

B-16.1.2 Reagents

B-16.1.2.1 Sulphuric acid — 95 percent (*m/m*).

B-16.1.2.2 Mercury

B-16.1.3 Procedure

B-16.1.3.1 Fill the apparatus with mercury so that when the tap *A* is open, mercury just passes into the cup-shaped funnel.

B-16.1.3.2 Crush the material so that it passes through 150-micron IS Sieve, and dry it to constant mass at 140 to 150°C. Transfer 0.45 to 0.50 g of the dried sample, accurately weighed, into the cup of the nitrometer. Shake the material in the cup with 0.5 ml of water. Introduce the solution into the bulb by lowering the levelling tube. Wash down the cup twice with 1 ml portion of water. Finally introduce 15 ml of sulphuric acid into the bulb. During all these operations take care that no air is introduced.

B-16.1.3.3 When the reaction subsides, shake vigorously to complete the reaction and allow the apparatus to attain room temperature. Adjust the level of the mercury column in the levelling tube so that its height is above the level of mercury in the burette by an amount equal to one-seventh of the height of the acid column present in the burette.

B-16.1.3.4 Read the volume of gas in the burette and also note the room temperature and atmospheric pressure.

B-16.1.4 Calculation

$$\begin{aligned} &\text{Potassium nitrate (as KNO}_3 \text{),} \\ &\text{percent by mass} \end{aligned} = \frac{0.1619 (p - a) V}{(273 + t) \times M}$$

where

- p* = atmospheric pressure in mm of mercury,
- a* = tension of aqueous vapour in mm at *t*° C,
- V* = volume in ml of gas in the burette,
- t* = room temperature in degree centigrade, and
- M* = mass in g of the dried sample taken for the test.

B-16.2 Kjeldahl Method

B-16.2.1 Determine nitrogen content in potassium nitrate by Kjeldahl method following the procedure prescribed in IS : 5194-1969*, and from that calculate the percentage by mass of potassium nitrate content.

*Method of determination of nitrogen Kjeldahl method.

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APPENDIX C

(Clause 5.1)

SAMPLING OF POTASSIUM NITRATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

C-1. GENERAL REQUIREMENTS OF SAMPLING

C-1.0 In drawing samples, the following precautions and directions shall be observed.

C-1.1 Samples shall not be taken in an exposed place.

C-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

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

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

C-1.6 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling and the date of sampling.

C-2. SAMPLING

C-2.1 Lot — In a single consignment of one grade of the material, all containers of the same 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 sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

C-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 from a lot shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

(Clause 2.1.1)

LOT SIZE (<i>N</i>)	SAMPLE SIZE (<i>n</i>)
(1)	(2)
Up to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

C-2.1.2 The containers shall be selected at random and to ensure randomness of selection reference may be made to IS : 4905-1968*. In case it is not available, the following procedure shall be followed:

'Arrange the containers in the lot in a systematic order and starting from any one, count them as 1, 2, , up to *r* and so on, where *r* is the integral part of N/n (*N* and *n* being the lot size and sample size respectively). Every *r*th container thus counted shall be withdrawn to constitute the sample.

C-3. PREPARATION OF TEST SAMPLES

C-3.1 From each of the containers selected, draw a small representative portion of the material not less than 200 g in mass.

C-3.2 Out of these portions, an equal quantity of the material shall be taken and mixed thoroughly to form a composite test sample of mass less than 0.5 kg. The composite test 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.

G-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 test sample. One of these three sets (each set containing one bottle representing each container sampled) shall be marked for the purchaser, another for the supplier and the third for the referee.

*Methods for random sampling.

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C-3.4 The referee test samples 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.

C-4. NUMBER OF TESTS

C-4.1 Tests for the determination of chlorides in the case of Grade 1 and nitrites in the case of Grade 2 shall be performed on each of the individual test samples.

C-4.2 Test for the determination of all the remaining characteristics given in 3 and Table 1 shall be carried out on the composite test sample.

C-5. CRITERIA FOR CONFORMITY

C-5.1 From the individual test results for chlorides in the case of Grade 1 the average (\bar{x}) and the range (R) of the test results shall be computed (range R is defined as the difference between the maximum and minimum values of test results).

C-5.1.1 A lot shall be declared as conforming to the specification requirements of chlorides in the case of Grade 1 and nitrites in the case of Grade 2 under the following conditions:

<i>Characteristic</i>	<i>Criterion for Conformity</i>
i) Chlorides	($\bar{x} + 0.6 R$) computed from relevant test results shall be less than or equal to 0.02
ii) Nitrites	All individual test results shall pass the test given in B-15.2.3

C-5.2 For declaring the conformity of the lot to the specification requirements of all other characteristics tested on the composite test sample, the test results shall meet the corresponding values specified.

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