

METRIC

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## DETAIL SPECIFICATION

POTASSIUM NITRATE  
(For use in ammunition)

This specification is approved for use by all Departments and Agencies of the Department of Defense.

## 1. SCOPE

1.1 Scope. This specification covers the requirements, examinations and tests for commercial grade Potassium Nitrate and is intended for use in ammunition.

1.2 Classes. The Potassium Nitrate is in the following classes:

- Class 1 - for use in black powder and chemical munitions.
- Class 2 - for use in pyrotechnic compositions.
- Class 3 - for use in propellants.

## 2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 or 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in section 3 or 4 of this specification, whether or not they are listed.

Comments, suggestions, or questions on this document should be addressed to:  
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2.2 Government documents.

2.2.1 Specification and standards. The following specifications and standards form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

## DEPARTMENT OF DEFENSE STANDARDS

|              |   |  |
|--------------|---|--|
| MIL-STD-1168 | - | Ammunition Lot Numbering and<br>Ammunition Data Card |
| MIL-STD-1916 | - | DOD Preferred Methods for Acceptance of<br>Product   |

(Copies of these documents are available online at <https://quicksearch.dla.mil/>.)

2.3 Non-government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

## ASTM International

|           |   |  |
|-----------|---|--|
| ASTM E11  | - | Standard Specification for Wire Cloth and<br>Sieves for Testing Purposes |
| ASTM E300 | - | Standard Practice for Sampling Industrial<br>Chemicals                   |

(Copies of these documents are available from [www.astm.org](http://www.astm.org).)

2.4 Order of precedence. Unless otherwise specified in this document or in the contract, in the event of a conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

## 3. REQUIREMENTS

3.1 Required inspections.

3.1.1 First article inspection. When specified (See 6.2), a sample shall be subjected to first article inspection in accordance with 4.3.

3.1.2 Conformance. A sample shall be subjected to conformance inspection in accordance with 4.4.

3.2 Moisture. The moisture content of the potassium nitrate shall be not greater than 0.2%.

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3.3 pH. The pH of the potassium nitrate solution shall be Neutral.

3.4 Grit. The potassium nitrate shall be free of grit.

3.5 Insoluble material. The insoluble material in the potassium nitrate shall be not greater than 0.1%.

3.6 Chemical requirements. The potassium nitrate shall comply with the chemical requirements specified in Table I when determined as specified in applicable paragraphs of section 4.

TABLE I. Chemical properties.

| Properties   | Class 1 | Class 2 | Class 3 | Verification Paragraph |
|--|---------|---------|---------|------------------------|
|  | Percent | Percent | Percent |                        |
| Chlorides, as potassium chloride (KCl), maximum (max.).....                          | 0.07    | 0.07    | 0.07    | 4.5.5.1                |
| Chlorates and perchlorates, as potassium perchlorate (KClO <sub>4</sub> ), max. .... | 0.5     | 0.5     | 0.5     | 4.5.5.2                |
| Iron and aluminum, as oxides, max. ....  | 0.5     | 0.5     | ----    | 4.5.5.3                |
| Calcium and magnesium, as oxides, max. ....  | 0.5     | 0.5     | 0.5     | 4.5.5.4                |
| Sodium, as oxide, max. ....  | 0.25    | 0.25    | ----    | 4.5.5.5                |
| Ammonium compounds.....  | None    | None    | ----    | 4.5.5.6                |
| Nitrogen, minimum (min.) .....   | 13.77   | 13.77   | 13.77   | 4.5.5.7                |

3.7 Odor. The potassium nitrate shall be odorless.

3.8 Color (crystals). The potassium nitrate shall be white.

3.9 Granulation.

3.9.1 Class 1. Class 1 potassium nitrate shall be furnished in lump form.

3.9.2 Class 2 and 3. Class 2 and 3 potassium nitrate shall comply with the granulation requirements shown in Table II, using sieves conforming to specification ASTM E11, when tested as specified in 4.5.6.

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TABLE II. Granulation requirement (Classes 2 and 3).

| U.S. Alternate Sieve Number | Percent, min. through specified sieve |         |
|-----------------------------|---------------------------------------|---------|
|                             | Class 2                               | Class 3 |
| 60                          | 99.9                                  | .....   |
| 80                          | .....                                 | 99.9    |
| 100                         | 40                                    | .....   |

3.10 Ammunition lot numbering. Ammunition lot numbers shall be assigned in accordance with MIL-STD-1168.

3.11 Foreign material. There shall be no foreign contaminants including dust, debris from machinery, or any other particle that is not intended to be in the material.

3.12 Workmanship. The potassium nitrate shall be uniform in quality and free from defects that would render it unsuitable for the intended use. Care shall be taken to protect the material from moisture and adverse environmental effects. This may be evidenced by leakage, loss of contents, objectionable dents, or corrosion. All required markings shall be correct, neat and legible.

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## 4. VERIFICATION

TABLE III. Requirement/verification cross reference matrix.

| Method of Verification<br>1 - Analysis<br>2 - Demonstration (end item test)<br>3 - Examination<br>4 - Test |                          | Classes of Verification<br>A - First Article Inspection<br>B – Conformance Inspection |                     |   |   |   |                    |   |
|--|--------------------------|---|---------------------|---|---|---|--------------------|---|
| Section 3 Requirements   | Description              | Section 4 Verifications   | Verification Method |   |   |   | Verification Class |   |
|  |                          |   | 1                   | 2 | 3 | 4 | A                  | B |
| 3.1.1  | First article inspection | 4.3   |                     |   | X | X | X                  |   |
| 3.1.2  | Conformance              | 4.4   |                     |   | X | X |                    | X |
| 3.2  | Moisture                 | 4.5.1   |                     |   |   | X | X                  | X |
| 3.3  | pH                       | 4.5.2   |                     |   |   | X | X                  | X |
| 3.4  | Grit                     | 4.5.3   |                     |   |   | X | X                  | X |
| 3.5  | Insoluble material       | 4.5.4   |                     |   |   | X | X                  | X |
| 3.6  | Chemical requirements    | 4.5.5   |                     |   |   | X | X                  | X |
| 3.7  | Odor                     | 4.4.2.1   |                     |   | X |   | X                  | X |
| 3.8  | Color                    | 4.4.2.1   |                     |   | X |   | X                  | X |
| 3.9  | Granulation              | 4.5.6   |                     |   |   | X | X                  | X |
| 3.10   | Ammunition lot numbering | 4.4.2.1   |                     |   | X |   | X                  | X |
| 3.11   | Foreign material         | 4.4.2.1   |                     |   | X |   | X                  | X |
| 3.12   | Workmanship              | 4.4.2.1   |                     |   | X |   | X                  | X |

4.1 Classification of inspection. The verification requirements specified herein are classified as follows.

- a. First article inspection (see 4.3)
- b. Conformance inspection (see 4.4)

4.2 Verification conditions. Unless otherwise specified, all verifications shall be performed in accordance with the test condition specified in the applicable test method document of the paragraphs in the specification.

4.3 First article inspection. When specified, a sample shall be subjected to first article inspection in accordance with Table IV.

4.3.1 First article quantity. The first article sample shall be not less than 455 grams of potassium nitrate from the first production batch produced by the contractor using the same production equipment, process procedures, and material supply sources as will be used in regular production.

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4.3.2 Rejection. The first article sample shall be rejected if any of the inspections/tests in Table IV fails to meet any of the requirements.

TABLE IV. First article inspection.

| Examination or Test      | No. of sample units | Requirement paragraph | Inspection method |
|--------------------------|---------------------|-----------------------|-------------------|
| Moisture content         | 1 sample            | 3.2                   | 4.5.1             |
| pH                       | 1 sample            | 3.3                   | 4.5.2             |
| Grit content             | 1 sample            | 3.4                   | 4.5.3             |
| Insoluble material       | 1 sample            | 3.5                   | 4.5.4             |
| Chemical requirements    | 1 sample            | 3.6                   | 4.5.5             |
| Odor                     | 1 sample            | 3.7                   | Smell/4.5.7       |
| Coloration               | 1 sample            | 3.8                   | Visual/4.5.7      |
| Granulation              | 1 sample            | 3.9                   | 4.5.6             |
| Ammunition lot numbering | 1 package           | 3.10                  | Visual            |
| Foreign material         | 1 sample            | 3.11                  | Visual/4.5.7      |
| Workmanship              | 1 sample            | 3.12                  | Visual/4.5.7      |

4.4 Conformance inspection.

4.4.1 Lot formation. Inspection lots shall comply with the lot formation provisions of MIL-STD-1916. A lot of potassium nitrate shall consist of material from batches or blending operations that is subjected to the same processing operations and conditions, produced by one manufacturer, in accordance with the same specification, or same specification revision, under one set of operating instructions.

4.4.2 Classification of characteristics.

a. Sampling for conformance. Inspection sampling requirements for Critical, Major, and Minor characteristics are as defined in MIL-STD-1916. In addition to the sampling requirements stated herein, unless otherwise specified, Inspection Level IV shall be used for all characteristics defined as Majors and Inspection Level II for all Minor characteristics; Critical characteristics shall be addressed in accordance with MIL-STD-1916.

b. Sampling for chemical/physical requirements. Ten percent of the containers in the lot shall be sampled, but in no case more than 10 nor less than 3 containers shall be selected so as to be representative of the lot. If there are less than 3 containers in the lot, all the containers shall be sampled. The material from each container shall be mixed to a uniform consistency throughout, and approximately 225 grams (g) of it shall be taken from each selected container in accordance with ASTM E 300. These primary samples shall be placed in separate airtight containers, labeled so that the container from which the sample were taken can be identified. A composite sample, of approximately 225 grams, shall be made from equal portions of the primary samples. The composite samples shall be thoroughly mixed and placed in an airtight

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container, labeled to show the name of the material, manufacture, plant, contract or purchase order number, and lot size. All acceptance tests shall be made on the composite sample; failure to meet any requirement shall be cause for lot rejection. All primary samples shall be held for possible future examination should composite sample fail to comply with the requirements.

c. Conformance inspection. Conformance inspection shall be performed in accordance with paragraph 4.4.2.1. For all conformance inspections the same sample specimen may be used for all non-destructive examinations or tests.

d. Rejection. The lot shall be rejected if any of the inspections/tests in 4.4.2.1 fails to meet any of the requirements.

| 4.4.2.1                     | Examination or test      | No. of sample units | Requirement paragraph | Inspection method |
|-----------------------------|--------------------------|---------------------|-----------------------|-------------------|
| <u>Critical</u>             | None                     |                     |                       |                   |
| <u>Major</u>                |                          |                     |                       |                   |
| 101                         | Moisture content         | 1 sample            | 3.2                   | 4.5.1             |
| 102                         | pH                       | 1 sample            | 3.3                   | 4.5.2             |
| 103                         | Grit content             | 1 sample            | 3.4                   | 4.5.3             |
| 104                         | Insoluble material       | 1 sample            | 3.5                   | 4.5.4             |
| 105                         | Chemical requirements    | 1 sample            | 3.6                   | 4.5.5             |
| 106                         | Odor                     | 1 sample            | 3.7                   | Smell/4.5.7       |
| 107                         | Coloration               | 1 sample            | 3.8                   | Visual/4.5.7      |
| 108                         | Granulation              | 1 sample            | 3.9                   | 4.5.6             |
| 109                         | Foreign material         | 1 sample            | 3.11                  | Visual/4.5.7      |
| <u>Minor</u>                |                          |                     |                       |                   |
| 201                         | Ammunition lot numbering | Level II            | 3.10                  | Visual            |
| 202                         | Workmanship              | Level II            | 3.12                  | Visual            |
| Note. Sampling see 4.4.2.b. |                          |                     |                       |                   |

#### 4.5 Testing.

4.5.1 Determination of moisture. Dry an accurately weighed portion of approximately 10 grams (g) of the sample in a tared dish at  $150 \pm 2$  degrees Centigrade ( $^{\circ}\text{C}$ ) to constant weight, cool in a desiccator and weigh. Calculate the loss in weight as percent moisture.

$$\text{Percent moisture} = 100 * \left( \frac{B - A}{B} \right)$$

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Where:

A = Dried sample

B = Original sample

4.5.2 Determination of pH. Dissolve a weighed portion of approximately 1 g of the sample in 20 milliliters (mL) of neutral distilled water and determine if sensitive blue litmus paper is changed in color within 3 minutes, when immersed in the solution. The blue litmus paper shall be considered to be sufficiently sensitive if it shows a distinct reddish coloration, after being immersed for 3 minutes in .0001 normal (N) hydrochloric or sulfuric acid.

Using the solution from the acidity test determine if sensitive red litmus paper is changed in color within 3 minutes, when immersed in the solution. The red litmus paper shall be considered sensitive enough if it shows a distinct bluish coloration, after being immersed for 3 minutes in .0001 N sodium hydroxide. The sample shall be considered neutral if the litmus paper does not change color.

4.5.3 Determination of grit. Dissolve an approximately 25 g portion of the sample in water, filter through a medium porosity filter, and wash thoroughly. Transfer any material retained on the filter to a smooth glass slide. Press and rub the material with a smooth steel spatula against the glass slide. The presence of grit is indicated by the persistence of a scratching noise.

4.5.4 Determination of insoluble material. Dissolve a weighed portion of approximately 10 g of the sample in 100 mL of boiling distilled water and filter the solution through a tared filtering crucible. Wash the insoluble residue on the filter with hot water. Dry the crucible and contents at 100°C to 105°C for 3 hours, cool in a desiccator, and weigh. Calculate the increase in weight as a percent insoluble material.

$$\text{Percent insoluble material} = 100 * \left( \frac{A - B}{C} \right)$$

Where:

A = weight of residue and crucible, in g

B = tared weight of crucible, in g

C = weight of sample, in g

#### 4.5.5 Chemical requirement.

4.5.5.1 Determination of chlorides. Dissolve an accurately weighed portion of approximately 5 g of the sample in 100 mL of distilled water and make the solution slightly acidic with nitric acid. Add a few drops of approximately 0.25N silver nitrate solution. If more than a faint opalescence results, add more silver nitrate solution until precipitation is complete. Boil for 2 minutes with constant stirring, and allow the precipitate to settle overnight in a dark



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place. Decant the solution through a tared filtering crucible. Wash the precipitate four times by decantation, using 20 mL portions of water containing 1 mL of concentrated nitric acid and 2 drops of silver nitrate solution per 100 mL. Transfer the precipitate to the crucible with 20 mL of the same wash water, wash it with 20 mL of 1 percent nitric acid solution and with two 20 mL portions of distilled water. Dry the crucible and precipitate for 4 hours at 135°C, cool in a desiccator, and weigh. Calculate the percent chlorides, as potassium chloride KCl, as follows:

$$\text{Percent KCl} = \frac{52A}{W}$$

Where:

A = weight of precipitate, in g

W = weight of sample, in g

4.5.5.2 Determination of chlorates and perchlorates. Transfer an accurately weighed portion of approximately 2 g of the sample to a platinum crucible, and mix with approximately 10 g of anhydrous sodium carbonate. Cover the mixture with a layer of sodium carbonate, fuse, and cool. Dissolve the fused mass in distilled water, render the solution slightly acid with nitric acid, and proceed with the precipitation as described in paragraph 4.5.5.1. Calculate the percent total chlorates and perchlorates, as Potassium perchlorate  $\text{KClO}_4$ , as follows:

$$\text{Percent KClO}_4 = 97 \frac{(A - 0.019 \times B \times W)}{W}$$

Where:

A = weight of precipitate, in g

B = percent KCl as determined in 4.5.5.1

W = weight of sample, in g

4.5.5.3 Determination of iron and aluminum (applicable to class 1 and 2 only). Dissolve a weighed portion of approximately 5 g of the sample in distilled water and filter the solution. Acidify the filtrate with nitric acid, add a slight excess of ammonium hydroxide, and boil. Catch the precipitate on the ashless filter paper and wash with hot distilled water. Retain the filtrate and washings for further analysis. Ignite the filter paper and precipitate, in a tared porcelain crucible, to constant weight. Cool the crucible and contents in a desiccator, and weigh. Calculate the increase in weight as percent iron and aluminum oxides.

$$\text{Percent Fe and Al oxides} = 100 \frac{C - D}{E}$$

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Where:

C = weight of precipitate and crucible, in g

D = weight of tared crucible, in g

E = weight of original sample (approx. 5 g), in g

4.5.5.4 Determination of calcium and magnesium. Dilute the combined filtrate and washings from the determination of iron and aluminum (see 4.5.5.3) to approximately 200 mL and boil. Evaporate the filtrate to a volume of about 20 mL, filter, and add 15 mL of 9N ammonium carbonate solution, and 15 mL of 95 percent ethyl alcohol. Allow the mixture to stand for at least 30 minutes, with frequent stirring. Filter through paper and wash the precipitate with 9N ammonium carbonate solution. Dissolve the precipitate in dilute hydrochloric acid, dilute to approximately 50 mL, boil, and add 1 mL of saturated ammonium oxalate solution. Allow the solution to stand for 12 hours, filter through a tared filtering crucible, and wash the precipitate with hot water containing ammonium oxalate. Ignite the precipitate, gently at first and then with a blast lamp, cool the crucible and contents in a desiccator, and weigh the anhydrous calcium oxalate precipitate. Calculate the increase in weight as percent calcium oxide.

$$\text{Percent Calcium oxide} = \frac{44A}{W}$$

Where:

A = weight of calcium oxalate precipitate, in g

W = weight of sample, in g

Concentrate the filtrate from the calcium determination to approximately 50 mL, add 10 mL of a 10 percent solution of ammonium phosphate, and 20 mL of a 10 percent solution of ammonia. Allow the solution to stand for 4 or 5 hours, filter through a tared filtering crucible, and wash with a 2.5 percent solution of ammonia. Dry the precipitate and ignite to constant weight with a blast lamp. Cool the crucible and contents in a desiccator, and weigh the anhydrous magnesium oxalate precipitate. Calculate the increase in weight as a percent magnesium oxide, as follows:

$$\text{Percent Magnesium oxide} = \frac{36A}{W}$$

Where:

A = weight of magnesium oxalate precipitate, in g

W = weight of sample, in g

4.5.5.5 Determination of sodium (applicable to Class 1 and 2 only).

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4.5.5.5.1 Magnesium uranyl acetate reagent. Prepare solution A by dissolving 90 g of uranyl acetate,  $\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ , and 60 mL of glacial acetic acid in sufficient distilled water to make a volume of 1 liter by heating to  $70^\circ\text{C}$  and stirring until solution is complete. Prepare solution B by dissolving 600 g of magnesium acetate,  $\text{Mg}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$ , in 60 mL of glacial acetic acid and sufficient distilled water to make a volume of 1 liter by heating to  $70^\circ\text{C}$ , and stirring until solution is complete. Mix together solutions A and B while at  $70^\circ\text{C}$  and cool the mixture to  $20^\circ\text{C}$ . After allowing the mixture to stand at this temperature for at least 2 hours, filter it through a dry filter paper into an amber-colored bottle. Store the bottle containing the solution where it is not exposed to direct sunlight. If a precipitate appears on standing, filter the solution again prior to use.

4.5.5.5.2 Alcohol wash liquid. Prepare an alcohol wash liquid as follows: Shake 1 mL of 1 percent sodium chloride solution with 25 mL of magnesium uranyl acetate reagent. Catch the precipitate on a filter paper and wash thoroughly with 95 percent ethyl alcohol. Mix the precipitate with 1 liter of 95 percent ethyl alcohol and allow to stand for 1 hour, with frequent shaking. Filter the solution and use the filtrate as a wash liquid.

4.5.5.5.3 Procedure. Transfer a weighed portion of approximately 0.2 g of the sample to a 150 mL beaker. Dissolve it in 5 mL of distilled water and cool the solution to  $20^\circ\text{C}$ . Add 100 mL of the magnesium uranyl acetate reagent which has previously been cooled to  $20^\circ\text{C}$ . While maintaining the temperature at  $20^\circ\text{C}$ , rapidly stir the contents of the beaker for  $\frac{1}{2}$  hour by means of a mechanical stirrer. Transfer the precipitate to a tared filtering crucible, using a total of 25 to 30 mL of the wash liquid for transferring and washing the precipitate. Dry the crucible and precipitate at  $100^\circ\text{C}$  to  $110^\circ\text{C}$  for  $\frac{1}{2}$  hour, cool in a desiccator, and weigh. Calculate the weight of the magnesium sodium uranyl acetate,  $\text{MgNa}(\text{UO}_3)_3(\text{C}_2\text{H}_3\text{O}_2)_9 \cdot 6\frac{1}{2}\text{H}_2\text{O}$ , as percent sodium oxide, as follows:

$$\text{Percent sodium oxide} = \frac{2.06A}{W}$$

Where:

A = weight of precipitate, in g

W = weight of sample, in g

4.5.5.6 Determination of ammonium compounds (applicable to class 1 and 2 only). Transfer an accurately weighed portion of approximately 1.0 g of the sample to a 150 mL beaker, dissolve in 25 mL of distilled water, and add sufficient excess of concentrated sodium hydroxide solution to render the potassium nitrate solution alkaline. Heat the mixture and test the vapors with a moist strip of red litmus paper of such sensitivity that it changes color in 30 seconds,

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when immersed in 0.0005N alkali and stirred continuously. Care should be taken that no spray from the solution touches the test paper. Note if any ammonium compounds are present in the sample, as indicated by the paper becoming blue in color.

#### 4.5.5.7 Determination of nitrogen.

##### 4.5.5.7.1 Standardization of the nitrometer.

4.5.5.7.1.1 Absolute method. Assemble the nitrometer (DuPont or equivalent) after thoroughly cleaning and drying all parts. The various parts, with the exception of the reservoir bulbs, are completely filled with mercury. Place 25 mL of 94.5 +/- 0.5 percent sulfuric acid in the generating bulb, draw air into the bulb to nearly the level of the lower shoulder by lowering the mercury reservoir and opening both stopcocks. Close both stopcocks and shake vigorously for 3 minutes. Connect the generating bulb into the compensating tube by adjustment of the two mercury reservoirs, keeping open the stopcock of the measuring tube. Continue the introduction of the air until the mercury in the reading tube is approximately at the 12.50 mark. Seal the compensating tube by means of a small blowpipe flame. Desiccate similarly, in the generating bulb, another portion of the air and transfer this to the reading tube until the mercury in the reading tube, compensating tube, and their mercury reservoirs is at the same level, the mercury in the reading tube being approximately at the 12.50 mark. (It is not necessary that these gas volumes be exact or equal.) Fill a small U tube manometer with sulfuric acid and attach to the reading tube. Allow the apparatus to come to constant temperature and adjust the mercury levels in the reading tube, compensating tube, and their mercury reservoirs so that the air in the reading tube is under atmospheric pressure, as shown by the acid levels in the arm of the manometer tube. Take the reading on the graduated scale of the calibrated reading tube, and at the same time note the temperature and barometric pressure. From these readings calculate the volume of air at 20°C and 760 millimeter (mm) pressure in the reading tube, as follows:

$$\frac{P_0 V_0 f_0}{T_0} = \frac{P_1 V_1 f_1}{T_1} \implies V_0 = \frac{P_1 V_1 T_0 f_1}{P_0 T_1 f_0}$$

$$V_0 = \frac{P_1 V_1 293(1 - 0.00018T_1)}{760(273 + T_1)(1 - 0.00018 * 20)} = \frac{0.3869 P_1 V_1 (1 - 0.00018T_1)}{273 + T_1}$$

Where:

$P_i$  = Pressure at standardized or elevated conditions  $i$ , mm Hg

$V_i$  = Volume at standardized or elevated conditions  $i$ , mL

$T_i$  = Temperature at standardized or elevated conditions  $i$ , °C

$f_i$  = Correction factor at standardized or elevated conditions  $i$

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Note that  $T_1$  should be approximately  $70^\circ\text{C}$

0.00018 = coefficient of expansion of mercury in glass to calculate  $f_i$

Adjust the heights of the reading tube, compensating tube, and their reservoirs so that, with the mercury in the reading and compensating tubes on a level, the mercury in the reading tube marks the calculated volume of the air. Paste a strip of paper on the tube so as to mark the height of the mercury in the compensating tube. Consider this as the standard volume of air with which every volume of gas, to be measured, is compared. (Note. The use of dry air tends to form a scum around the reading level of the compensating tube, thus requiring frequent restandardization of the nitrometer. This scum is caused by reaction of the oxygen in the air with mercury, or by impurities. Dry nitrogen eliminates the condition and should be used, if available. A half drop of sulfuric acid will also prevent this scum.)

4.5.5.7.1.2 Potassium nitrate method. Recrystallize three times, from 95 percent alcohol, a sample of reagent potassium nitrate, and grind this to pass through a U.S. Alternate Sieve No. 100 IAW ASTM E11. After drying at  $150^\circ\text{C}$  for 2 hours, weigh an accurately measured portion of approximately 1.0 g into a small weighing bottle and transfer this portion to the cup of the generating bulb. Wash the potassium nitrate into the generating bulb by means of eight to ten portions of 94.5 +/- 0.5 percent nitrogen free sulfuric acid cooled to approximately  $5^\circ\text{C}$ , using a total volume of 25 mL of the acid. With the lower stopcock of the generating bulb open and the mercury reservoir low enough to maintain a slightly reduced pressure in the generating bulb, shake the sample until most of the gas has been generated. Adjust the mercury reservoir until the mercury drops to the lower shoulder of the generating bulb. Close the lower stopcock and shake the bulb vigorously for 3 minutes. Replace the bulb on the rack, open the lower stopcock, and allow the tube to stand for 5 minutes, the levels in the generating bulb and reservoir being approximately the same, having previously introduced desiccated air into the compensating tube as described in 4.5.5.7.1.1. Close the lower stopcock, and shake the generating bulb for 1 additional minute. Transfer the gas to the reading tube, and adjust the levels of the mercury in the reading and compensating tubes to approximately the same height. Allow the gas to stand for approximately 20 minutes, to permit equalization of the temperature. With a sulfuric acid U tube leveling device make careful adjustments, so that the mercury in both tubes is at the same level, and the mark in the reading tube is at the 13.85 mark (the percentage of nitrogen in potassium nitrate). Make a mark on the compensating tube to show the volume of nitric oxide gas liberated from exactly 1 g of potassium nitrate. (This mark may be made on a strip of gummed paper attached to the compensating tube.)

(Note 1. In order to avoid any doubt regarding the purity of the potassium nitrate, it is desirable to check the standardization obtained by use of potassium nitrate by means of the absolute method. If the agreement is within one or two hundredths of one percent, the potassium nitrate can be considered as of satisfactory quality and used in subsequent periodic standardizations. It

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has been found by actual experimentation that the solubility of nitric oxide in sulfuric acid, of the strength prescribed, is so small that no correction need be applied to bring the value in agreement with the mark set by the absolute method.)

(Note 2. If the nitrometer is standardized by both the absolute and potassium nitrate methods, the results may not be in agreement by 0.01 to 0.04 percent nitrogen due to expansion of the brass barometer scale. Such difference may be removed by applying the brass scale correction corresponding to the temperature (see 6.6).)

4.5.5.7.1.3 Procedure. Crush an accurately measured portion of approximately 3.0 g of the sample so that it will pass through a U.S. Alternate Sieve No. 100 IAW ASTM E11, and dry for 2 hours at 150°C. Accurately weigh approximately 1.0 g of the dried sample and transfer it to the cup of a standardized 5 part nitrometer (DuPont or equivalent). Measure into a small graduate, 25 mL of nitrogen free 94.5 +/- 0.5 percent sulfuric acid cooled to approximately 5°C. Wash out the weighing bottle into the nitrometer cup with 5 mL of the sulfuric acid. Lower the mercury reservoir sufficiently to maintain a reduced pressure in the generating bulb. Open wide the upper stopcock and open the lower stopcock sufficiently to draw the acid mixture into the generating bulb. Make successive 4 mL rinsings with the rest of the sulfuric acid until it has all been drawn in. Close the upper stopcock, leaving the lower one open, and adjust the mercury reservoir just low enough to maintain a slightly reduced pressure in the generating bulb. Shake the bulb gently until most of the gas has been generated. Lower the mercury reservoir until the level of the mercury in the generating bulb is at the height of the lower shoulder, then close the lower stopcock and shake vigorously for 3 minutes. Replace the bulb on the rack, open the lower stopcock, and adjust the mercury reservoir until the mercury in the generating bulb is at approximately the same height as the mercury in the reservoir. This will bring the pressure inside the generating bulb to approximately one atmosphere, and the solubility of the nitric oxide gas in the sulfuric acid will be normal. Close the lower stopcock and shake the bulb vigorously for an additional 1 minute. Transfer the gas to the measuring tube and adjust the levels of the mercury in the compensating and measuring tubes to approximately the same height. Let the gas stand for 20 minutes in order to permit equalization of the temperature of the gas in the two tubes, and adjust the mercury levels closely with a sulfuric acid U tube leveling device, the mercury level in the compensating tube being at the standard volume mark. Calculate the percent nitrogen as follows:

$$\text{Percent nitrogen} = \frac{R}{W}$$

Where

R = reading of the measuring tube.

W = weight of sample, in g

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4.5.6 Granulation. Nest the specified sieve or sieves (see Table II) on a bottom pan. Place a weighed portion of 100 g of sample on the upper sieve, cover the sieve and shake the assembly for 10 minutes by hand, or 5 minutes by means of a mechanical shaker, geared to produce 268 to 315 gyrations and 150 +/- 10 taps of the striker per minute. Weigh the amounts retained or passing through the sieves, and calculate to percentages as required.

4.5.7 Sample examination. Inspect residual quantity of material from composite sample for foreign material, color, and odor. The sample shall be examined with unaided eyes by personnel with normal vision or normal corrected vision.

## 5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in contract or order (see 6.2). When packaging of materiel is to be performed by DOD or in-house personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Service or Defense Agency, or within the military service's system command. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

## 6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory).

6.1 Intended use. Commercial-grade potassium nitrate is intended for use in ammunition.

6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number, and date of this specification.
- b. Class of material required (see 1.2).
- c. Packaging requirements (see 5.1).
- d. Requirements for First article samples (see 4.3).
- e. Provision for submission of first article samples.
- f. Requirements for submission of acceptance inspection equipment.
- g. Requirements for ammunition lot numbering.

6.3 Submission of contractor acceptance inspection equipment (AIE) designs for approval. Submit copies of designs as required to: Commander, U.S. Army Armaments Center, ATTN: FCDD-ACE-QMG, Picatinny, NJ 07806-5000. This address will be specified on the Contract Data Requirements List, DD Form 1423 in the contract.

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6.4 Equivalent test method approval. Prior approval of the contracting officer is required for use of equivalent test methods. A description of the proposed method should be submitted to the Technical Agency (see 6.3). This description should include but not be limited to the procedures used, the accuracy and precision, and drawings of any special equipment required.

6.5 Batch. A batch is defined as that quantity of material which has been subjected to some unit chemical or physical mixing process intended to make the final product substantially uniform.

6.6 Brass scale correction. An explanation of the procedure for making a brass scale correction will be found in Lange's Handbook of Chemistry, "Barometry and Barometric Corrections" (see note 2 in paragraph 4.5.5.7.1.2).

6.7 Subject term (key word) listing.

Black powder  
Propellant  
Pyrotechnic

6.8 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

Custodians:  
Army – AR  
Navy – OS  
Air Force – 170

Preparing Activity:  
Army – AR  
(Project 6810-2014-054)

Review Activities:  
Army – AV,TE  
Navy – AS, SH  
Air Force – 11

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at <https://assist.dla.mil>.