

MIL-A-50460A (MU)
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SUPERSEDING
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MILITARY SPECIFICATION

AMMONIUM NITRATE, PRILLED (FOR USE IN AMMUNITION)

1. SCOPE

1.1 This specification covers a high density phase-stabilized grade of prilled ammonium nitrate (NH_4NO_3) for use in explosives (see 6.4).

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

L-P-378 - Plastic Sheet and Strip Thin Gauge,
Polyolefin

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables
for Inspection by Attributes
(ABC-STD-105)
MIL-STD-109 - Quality Assurance Terms and
Definitions
MIL-STD-129 - Marking for Shipment and
Storage
MIL-STD-1168 - Lot Numbering of Ammunition
MIL-STD-1235 - Single and Multilevel Continuous
Sampling Procedures and Tables
for Inspection by Attributes

FSC: 1376

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(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the Contracting Officer.)

PUBLICATIONS

AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM Designation E203-64 - Water Using Karl
Fischer Reagent

ASTM Designation E300-70 - Recommended Practice
for Sampling Industrial
Chemicals

(Application for copies should be addressed to American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania, 19103)

2.2 Other publications. The following document forms a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

CODE OF FEDERAL REGULATIONS

Title 49 - Transportation, Parts 1-199

(The Interstate Commerce Commission Regulations are now a part of the Code of Federal Regulations, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402. Orders for the above publications should cite: "49 CFR 1-199 (latest revision)".)

3. REQUIREMENTS

3.1 Material. The ammonium nitrate shall be in the form of high density phase-stabilized prills having no coating (see 6.6).

3.2 Chemical Requirements. The ammonium nitrate shall conform to the chemical requirements of Table I when tested in accordance with applicable paragraphs of 4.4.

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TABLE I

Property	Requirement	Test Method
Moisture, %	0.15 max.	4.4.1
Ether-Soluble Material, %	0.05 max.	4.4.2
Water-Insoluble Material of ether-extract, %	0.10 max.	4.4.3
Water-Insoluble Material:		
a. Retained on a 250 micron (No. 60) sieve, %	0.00	4.4.4
b. Retained on a 125 micron (No. 120) sieve, %	0.01 max.	4.4.4
Acidity, as nitric acid, %	0.02 max.	4.4.5
Nitrites, %	None	4.4.6
Chlorides, as ammonium chloride, %	0.02 max.	4.4.7
Phosphates, as diammonium phosphate, %	0.21 \pm 0.04	4.4.8
Sulfates, as diammonium sulfate, %	0.007 to 0.014	4.4.9
Boric acid, %	0.14 \pm 0.03	4.4.10
Ammonium nitrate, %	98.5 min.	4.4.11
Density: Particle, g/ml	1.50 min.	4.4.12
Bulk, g/ml	0.80 min.	4.4.13
Bulk, lbs/cu. ft.	50.0 min	4.4.13
pH	5.9 \pm 0.2	4.4.14

3.3 Granulation. The high density phased-stabilized prilled ammonium nitrate shall conform to the granulation requirements of Table II when determined in accordance with 4.4.15.

TABLE II

Distribution	Percent by Weight
Through a 3360 micron (No. 6) sieve, (min)	99.0
Retained on a 1680 micron (No. 12) sieve	50.0 - 85.0
Retained on a 840 micron (No. 20) sieve, (min.)	97.0
Through a 500 micron (No. 35) sieve, (max.)	0.5

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3.4 Water-vapor transmission rate (WVTR). The plastic liner of the multi-wall paper bag or the all-plastic bag, including seams, shall meet a maximum WVTR of 0.65 grams/100 in sq/24 hours when tested as specified in paragraph 4.4.16.

3.5 First Article Testing. This specification makes provisions for first article testing. Submission of the first article quantity by the contractor shall be as specified in the contract.

3.6 Workmanship. The prilled ammonium nitrate furnished under this specification shall be free of dirt, chips and other foreign material.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements. Reference shall be made to MIL-STD-109 in order to define the terms used herein.

4.1.1 Submission of product. At the time the completed lot of product is submitted to the Government for acceptance the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product being submitted:

a. A statement that the lot complies with all requirements and quality assurance provisions specified in this specification.

b. Specification number and date, together with an identification and date of changes.

c. Certificates of analysis on all material used directly by the contractor when such material is controlled by Government specifications, shall be made available upon request by the contracting officer.

d. Quantity of product in the lot.

e. Date submitted.

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The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

4.2 First article inspection

4.2.1 Submission. Prior to initiation of regular production the contractor shall submit a first article sample consisting of 5 lbs of prilled ammonium nitrate in accordance with instructions issued by the Contracting Officer for evaluation in accordance with paragraph 4.2.2. All samples submitted shall have been produced by the Contractor using the same production processes, procedures, and equipment as will be used in fulfilling the contract. All materials, including packaging and packing, shall be obtained from the same sources of supply as will be used in regular production. The sample shall be accompanied by certificates of analysis. A first article quantity, or portion thereof, as directed by the Contracting Officer, shall also be submitted whenever there is a lapse in production for a period in excess of 90 days, or whenever a change occurs in manufacturing process, material used, drawing, specification or source of supply as to significantly affect product uniformity as determined by the Government. Prior to submission, the contractor shall inspect the sample to the degree necessary to assure that it conforms to the requirements of the contract and submit a record of this inspection with the sample. A sample containing known defects will not be submitted unless specifically authorized by the Contracting Officer (see 6.1).

4.2.2 Inspections to be performed. The sample will be subjected by the Government to any or all of the examinations or tests specified in 4.3 and 4.4 of this specification and any or all requirements of the applicable drawings.

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4.2.3 Rejection. If any sample fails to comply with any of the applicable requirements, the first article quantity shall be rejected. The Government reserves the right to terminate its inspection upon any failure of a sample to comply with any of the stated requirements.

4.3 Inspection provisions

4.3.1 Lot formation. A lot shall consist of one or more batches of ammonium nitrate produced by one manufacturer in accordance with the same specification, or same specification revision under one continuous set of operating conditions. Each batch shall consist of that quantity of ammonium nitrate that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogeneous. The product shall be submitted for inspection in accordance with MIL-STD-105. The criteria and procedures for the assignment of lot numbers shall be in accordance with MIL-STD-1168.

4.3.2 Sampling for test 4.4.1 through 4.4.15. Approximately 5 pounds of ammonium nitrate shall be selected from each batch to be sampled using ASTM Procedure E300-70 for solids. Sample shall be selected for inspection in accordance with MIL-STD-1235, CSP-1 Plan, Inspection Level II, AQL 6.5%. If any sample fails to meet any test requirement the batch represented by the sample shall be rejected. All batches produced between the time that the last batch was tested and accepted and the batch which failed shall be tested in accordance with the applicable methods given in paragraph 4.4. If any of these batches fail to meet any of the test requirements, that batch shall also, be rejected. In addition, after any failure of a batch the contractor will return to 100% inspection until "1" successive batches are accepted as required by MIL-STD-1235. The classification and code number shall be as given in Table III.

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TABLE III
CLASSIFICATION OF DEFECTS

Category	Defect	Code No. (see 6.2)
Moisture (see 6.5)	Major	03001
Ether-soluble material	Major	04001
Water-insoluble material of ether-extract	Major	05001
Water-insoluble material retained on a 250 micron and 125 micron sieve	Major	06001
Acidity, as nitric acid	Major	07001
Nitrites	Major	08001
Chlorides, as ammonium chloride	Major	09001
Phosphates, as diammonium phosphate	Major	10001
Sulfates, as diammonium sulfate	Major	11001
Boric acid	Major	12001
Ammonium nitrate	Major	13001
Density, particle	Major	14001
Density, bulk	Major	15001
pH	Major	16001
Granulation	Major	17001
Water-vapor transmission rate	Major	18001

4.3.3 Examination. Sampling plans and procedures for the following classifications of defects shall be in accordance with MIL-STD-105 (ABC-STD-105). Contractor's sampling plans, if used, shall be approved by the Government and shall provide as a minimum, the protection afforded the Government by the sampling plans in MIL-STD-105. Continuous sampling plans in accordance with MIL-STD-1235 may be used if approved by the procuring activity. Also, at the option of the procuring activity, AQL's and sampling plans may be applied to the individual characteristics listed, using an AQL of 0.40 percent for each Major defect and an AQL of 0.65 percent for each Minor defect.

4.3.3.1 Bag, prior to loading

Categories	Defects	Method of Inspection	Code No.
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Critical: None defined.

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Major:	AQL 0.65 percent		
101.	Thickness of liner incorrect	Gage(CD)	01001
102.	Seam insecurely bonded	Visual/Manual	01002
103.	Bag contains hole or cut	Visual	01003
104.	Presence of foreign matter	Visual	01004

Minor:	AQL 0.65 percent		
201.	Evidence of poor workmanship	Visual	01005

4.3.3.2 Bag, after loading

Categories	Defects	Method of Inspection	Code No.
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Critical: None defined.

Major:	AQL 0.65 percent		
101.	Bag split or punctured permitting contents to spill	Visual	02001
102.	Closures incomplete or mislocated	Visual/Manual	02002
103.	Label missing	Visual	02003
104.	Weight incorrect	Balance(CD)	02004

Minor:	AQL 1.50 percent		
201.	One or more plies torn	Visual	02005
202.	Marking misleading or unidentifiable	Visual	02006
203.	Evidence of poor workmanship	Visual	02007

4.3.3.3 Water-vapor transmission rate. A sample of 10 bags of ammonium nitrate shall be selected at random for each 10,000 bags produced. Failure of one or more bags to comply with the applicable requirement in paragraph 3.4 shall be cause for rejection of the lot.

4.3.4 Inspection equipment. The inspection equipment required to perform inspection prescribed in this section is identified in the Examination (4.3.3) and Test Method (4.4) paragraphs herein. See 6.3 for details concerning responsibilities for inspection equipment design and approval.

4.4 Test Methods and Procedures

4.4.1 Moisture. The moisture shall be determined in accordance with ASTM Method E203-64 except that 7 to 9 grams of sample shall be added to 1:1 mixture of pyridine and methanol. Karl Fischer reagent in the form of the stabilized single solution is preferred for use in this determination, but the reagent which is divided into solution A, containing pyridine, sulfur dioxide, and methanol, and solution B, containing iodine dissolved in methanol may be used.

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4.4.1.1 Calculate the moisture content as follows:

$$\text{Moisture, \%} = A - (B \times 0.874)$$

Where: A = Percent apparent water determined in 4.4.1.

B = Percent boric acid determined in 4.4.10.

4.4.2 Ether-soluble material. Dry a clean 150 milliliter (ml) beaker in an oven at 100°C for one hour, cool in a desiccator and weigh to the nearest mg. Weigh to the nearest mg approximately 25 g of the sample and transfer to the thimble of a soxhlet extractor containing anhydrous ether. Extract the sample for 2 hours. Save the extracted sample for the water-insoluble determination (4.4.3). Evaporate the ether extract in the extraction flask to a volume of approximately 50 ml on a steam bath and transfer to the weighed beaker. Rinse out the extraction flask with several 5-ml portions of ether and add to the main extract in the beaker. Cover the beaker with a ribbed watch glass and evaporate on a steam bath. After the solvent is removed, heat the beaker and residue in an oven at 100°C for 1 hour, cool in a desiccator, and weigh to the nearest mg. Calculate the percent of ether-soluble material as follows:

$$\text{Percent ether-soluble material} = \frac{100A}{W}$$

Where:

A = weight of residue from sample, in g

W = weight of sample used, in g

4.4.3 Water-insoluble material. Transfer the ether-extracted sample (see 4.4.2) to a beaker, dissolve in hot water, and filter through a tared filtering crucible. Wash the insoluble residue in the crucible with hot water until it is free from nitrate. Dry to constant weight at 100°C, cool in a desiccator, and weigh to the nearest mg. Calculate the percent of water-insoluble material as follows:

$$\text{Percent water-insoluble material} = \frac{100A}{W}$$

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Where:

A = Weight of residue, in g

W = weight of sample, used, in g

4.4.4 Water-insoluble material retained on a 250 micron (No. 60) sieve and on a 125 micron (No. 120) sieve. Weigh to the nearest 0.01 g approximately 100 g of the sample, transfer to a beaker, and dissolve in hot water. Pour the solution through a No. 60 sieve which is nested on top of a No. 120 sieve, and transfer any insoluble matter from the beaker to the upper No. 60 sieve by means of a jet of water. Wash the insoluble matter in the No. 60 sieve thoroughly by means of a jet of hot water. When no more insoluble matter passes through the sieve on to the No. 120 sieve, remove the No. 60 sieve and wash the insoluble material on the No. 120 sieve with hot water until no more insoluble matter passes through this sieve. Dry the two sieves and the residues remaining on them at 100°C for one hour. Transfer the dry residue from each sieve to a separate tared glass weighing dish. Weigh each dish to the nearest 0.01 g and calculate the percent insoluble material retained on the sieve as follows:

$$\text{Percent insoluble material retained on sieve} = \frac{100A}{W}$$

Where:

A = weight of material retained on sieve, in g

W = weight of sample, used in g

4.4.5 Acidity as nitric acid. Weigh to the nearest 0.01 g approximately 100 g of the sample and dissolve in 400 ml of water. Filter, add methyl red indicator, and titrate with 0.1 N sodium hydroxide. Calculate the acidity as follows:

$$\text{Acidity, as percent nitric acid} = \frac{6.3 \times N \times V}{W}$$

Where:

N = normality of the sodium hydroxide used.

V = volume of sodium hydroxide used to titrate sample
in ml

W = weight of sample, in g

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4.4.6 Nitrites

4.4.6.1 Metaphenylenediamine hydrochloride solution. Dissolve 0.5 g of metaphenylenediamine hydrochloride in 100 ml of distilled water. If the solution is colored when prepared, add some animal charcoal, shake thoroughly, and filter to decolorize.

4.4.6.2 Procedure. Dissolve a weighed portion of approximately 1 g of the sample in 20 ml of distilled water. Filter the solution before proceeding. Add 1 ml of a 10 percent by volume solution of sulfuric acid and 1 ml of metaphenylenediamine hydrochloride solution (see 4.4.6.1). No yellow or yellowish-brown color shall develop.

4.4.7 Chlorides as ammonium chloride. Weigh 5 g of the sample to the nearest 0.01 g and transfer to a 100 ml low form Nessler tube, add 50 ml of distilled water, and shake until solution is complete. Filter the solution before proceeding. Add 10 ml of a 10-percent solution by weight of silver nitrate and mix well. Make up to the mark with distilled water and again mix well. Simultaneously and in the same manner, prepare a standard containing 0.0010 g of ammonium chloride and 5.000 g reagent grade ammonium nitrate and the foregoing amount of silver nitrate. As soon as the silver nitrate solution is added compare the turbidities of the test and standard solutions. The turbidity of the sample tube shall not exceed that of the standard tube.

4.4.8 Phosphate, as diammonium phosphate

4.4.8.1 Apparatus. Spectrophotometer, Beckman DU or equivalent with cells; Burettes, 100 ml; Beakers, 250 ml; Volumetric flasks, 500 and 1000 ml; Pipets, 5 and 10 ml.

4.4.8.2 Chemicals required. Ammonium molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ Reagent grade; Sulfuric acid, concentrated, reagent grade; Sodium sulfite, anhydrous, reagent grade; Sodium meta bisulfite, reagent grade; 1-Amino-2-naphthol-4 sulfonic acid; Potassium dihydrogen phosphate (KH_2PO_4) reagent grade; Ammonium nitrate, anhydrous, reagent grade.

4.4.8.3 Preparation of solutions

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4.4.8.3.1 Ammonium molybdate solution. Dissolve 28 grams weighed to nearest 0.01 gram of $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ in about 250 ml of distilled water. Cautiously and with stirring add 225 ml of sulfuric acid. Cool and dilute to 1000 ml at 20°C.

4.4.8.3.2 Amino acid solution. Dissolve 2.5 grams of sodium sulfite, 1.25 grams of 1-amino -2-naphthol-4 sulfonic acid and 68.5 g sodium metabisulfite in 250 ml of distilled water. Warm the mixture, if necessary, to facilitate solution of components. Dilute the clear solution to 500-ml at 20°C.

4.4.8.3.3 Standard diammonium phosphate (DAP) solution. Dry a sample of KH_2PO_4 for one hour at 105°C and then allow to cool in a desiccator. Dissolve exactly 1.030 grams of this dried material in about 250 ml of distilled water, contained in a 1 liter volumetric flask, and dilute the solution to mark with additional distilled water. Dissolve 1.84 grams of ammonium nitrate in about 250 ml of distilled water, contained in a 1 liter volumetric flask. Add a 10 ml aliquot of the KH_2PO_4 solution (above), and dilute to mark with distilled water. This solution contains 1.84 mg of ammonium nitrate and 0.01 mg KH_2PO_4 (equivalent to 0.01 mg DAP) per ml of solution and is referred to hereinafter as the DAP standard. (It should be noted that in the determination of DAP in ammonium nitrate, the final aliquot for analysis contains 0.184 g of ammonium nitrate which may offer some interference with color development. To offset this effect, the same quantity of ammonium nitrate is added to the solution used for standardization as is present in the above noted final aliquot).

4.4.8.3.4 Ammonium nitrate stock solution. Dissolve exactly 1.84 grams of ammonium nitrate (reagent grade) in 250 ml of distilled water contained in a 1 liter volumetric flask and dilute to mark with distilled water (see 4.4.8.3.5)

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4.4.8.3.5 Preparation of standard curve for DAP. For the establishment of a standard curve use the quantities shown in column 1 and column 2 of Table IV. Use 100 ml burettes to deliver the volume of solutions to 250 ml beakers. Column three shows the quantity of DAP present (based on 1.030 g KH_2PO_4) in the aliquot of the DAP standard solution. To each solution contained in 250 ml beaker add 10.0 ml ammonium molybdate solution and 5.0 ml of amino acid solution. Mix the resulting solution thoroughly, allow color to develop for 10 minutes and read the absorbance within 15 minutes using a spectrophotometer set at 730 mu. Use distilled water as a blank. Plot the absorbance versus the respective weight of DAP. The plot should afford a straight line.

TABLE IV

Stock solution of <u>Ammonium nitrate, ml</u>	DAP Standard <u>solution, ml</u>	DAP Present, <u>gram</u>
100.0	0.0	0.0000
90.0	10.0	0.0001
80.0	20.0	0.0002
60.0	40.0	0.0004
40.0	60.0	0.0006
0.0	100.0	0.0010

4.4.8.4 Procedure. Dissolve 4.6 grams of the sample to be tested, weighed to the nearest 0.01g, in about 250 ml of distilled water. Filter the solution through a Whatman No. 42 filter paper or equivalent. Transfer the solution to a 500 ml volumetric flask, and make up to mark with distilled water (at 20°C). Transfer a 20 ml aliquot of this solution to a 250 ml beaker and dilute to exactly 100.0 ml with distilled water. To the diluted solution add a 10 ml aliquot of the molybdate solution and a 5 ml

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aliquot of the amino acid solution. The volume should total exactly 115.0 ml. Mix thoroughly, allow color to develop 10 minutes, read absorbance within 15 minutes using a spectrophotometer set at 730 mu. Determine the weight of DAP in aliquot from the standard curve for DAP and calculate the percent DAP in sample as follows:

$$\% \text{ DAP} = \frac{25}{W} \frac{A \times 100}{W}$$

Where: A = weight of DAP in aliquot, in grams

W = weight of sample, in grams

Determine the absorbance of a mixture of 100 ml distilled water, 10.0 ml of molybdate solution and 5 ml amino acid solution. The absorbance value obtained should check with that obtained for the preparation of the standard chart for DAP.

4.4.9 Sulfates

4.4.9.1 Diammonium sulfate (DAS). Weigh 10 grams of sample to the nearest 0.01 g and dissolve in 500 ml of distilled water. Filter the solution before proceeding. Transfer a 40 ml aliquot of this solution to a 50 ml Nessler tube. Dilute to the mark with 0.1 N barium chloride solution.

4.4.9.2 Preparation of standard. Weigh exactly 0.740 g of CP barium chloride dihydrate and dissolve in 1000 ml of distilled water. Transfer a 10 ml aliquot of this solution to a 1000 ml volumetric flask and dilute to the mark with distilled water (at 20°C). This solution is used as a standard.

4.4.9.3 Procedure

4.4.9.3.1 Transfer 28 ml of the DAS Standard to a 50 ml Nessler tube and dilute to mark with 0.1N sulfuric acid. Compare this tube with the tube from 4.4.9.1. The turbidity of the sample shall not exceed that of the standard.

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4.4.9.3.2 Transfer 14 ml of the DAS standard to a 50 ml Nessler tube, add 20 ml distilled water, and dilute to the mark with 0.1 N sulfuric acid. Compare this tube with the tube from 4.4.9.1. The turbidity of the standard shall not exceed that of the sample.

4.4.10 Boric acid (H_3BO_3)

4.4.10.1 Apparatus. pH meter, with capability of reading to 0.01 pH unit; magnetic stirrer; beakers, 250 ml; burette, 25 ml.

4.4.10.2 Chemicals required. Sodium hydroxide solution, 0.1N and 1.6N; Sodium hydroxide standard solution, 0.02N; D-mannitol, powder; Formaldehyde, 37% by weight; Boric acid, USP powder; Ammonium nitrate, reagent grade, boric acid free; Hydrochloric acid solution, 2 to 3 N.

4.4.10.3 Standardization of 0.02N sodium hydroxide solution. Weigh 5 grams (to nearest 0.01g) of anhydrous reagent grade ammonium nitrate and 0.0100 gram boric acid. (If desired, instead of the weighed amount of boric acid use a 5.0 ml aliquot of a boric acid solution containing 0.2000 g boric acid in 100 ml water (made up to volume in a 100 ml volumetric flask). To avoid possible boric acid contamination use boron-free containers to store solution.) Transfer components into a 250 ml beaker and add 38 ml of water. (Add 33 ml of water if using 5.0 ml aliquot of boric acid solution). Allow the boric acid and ammonium nitrate to dissolve and treat the solution as described in 4.4.10.4.2. Calculate the boric acid titer for 0.02N sodium hydroxide solution as follows:

$$\text{Boric acid titer for 0.02N NaOH} = \frac{\text{gram of boric acid}}{\text{ml of 0.02N NaOH}}$$

(The boric acid titer should be checked as often as possible, at least every two or three sample runs).

4.4.10.4 Determination of boric acid in prilled ammonium nitrate.

4.4.10.4.1 Weigh 5 grams (to nearest 0.01g) of sample and transfer to a 250 ml beaker. Add 38 ml of distilled water, allow sample to dissolve and continue with 4.4.10.4.2.

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4.4.10.4.2 Add 50 ml of 1.6N NaOH and 12 ml of 37 percent formaldehyde solution. Stir the mix thoroughly until solution is complete and let solution stand for 5 minutes. Using a magnetic stirrer to agitate the dissolved sample, together with pH meter, adjust the pH of the solution to about 8.0 with dilute hydrochloric acid solution. Cool the solution to room temperature with iced water and readjust the pH to 8.20 with 0.1N sodium hydroxide and/or 0.02N sodium hydroxide (if pH happens to go beyond 8.20 use dilute HCl and NaOH solution to bring the pH to 8.20) Add 1.5 grams of D-mannitol and stir until solution is complete. Titrate the clear solution with standardized 0.02N sodium hydroxide solution to the pH of 8.20 (pH before addition of D-mannitol) and record the ml of 0.02N NaOH.

4.4.10.4.3 Calculate the percent boric acid as follows:

$$\text{Boric acid, \%} = \frac{\text{ml of 0.02N NaOH} \times \text{Boric acid titer for NaOH}}{\text{weight of sample}} \times 100$$

4.4.11 Ammonium nitrate. To 100 ml of distilled water in an Erlenmeyer flask, add 25 ml of a 37 percent by weight solution of formaldehyde and a few drops of phenolphthalein indicator solution. Neutralize with 0.15 N sodium hydroxide solution, add approximately 1 g of the sample, weighed to the nearest 0.1 mg. Titrate the solution with 0.15 N sodium hydroxide to an end point which persists for 30 seconds. (The sodium hydroxide may be standardized with standard reagent grade ammonium nitrate). Calculate the percent ammonium nitrate as follows:

$$\text{Percent (\%)} \text{ NH}_4\text{NO}_3 = \frac{8.005 \times V \times N}{W} - 1.2119^* \times \% \text{ DAP} - 1.3908^* \times \% \text{ DAS}$$

Where: V = Volume of sodium hydroxide solution used, in ml
N = Normality of sodium hydroxide solution used
W = Weight of sample used, in g

*1.2119 and 1.3908 are the conversion factors of DAP and DAS, respectively, to ammonium nitrate. (See 4.4.8 and 4.4.9)

4.4.12 Density, particle

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4.4.12.1 Density, particle. To a 1000 ml graduated cylinder add 500 ml of CP grade acetone and pour a weighed sample of 500 grams of ammonium nitrate into the acetone. As soon as the bubbles stop rising read the new volume and calculate the density as follows:

$$\text{Density, particle (g/ml)} = \frac{500\text{g}}{\text{increase in volume (ml)}}$$

4.4.13 Density, bulk

4.4.13.1 Density, bulk. Pour the sample into a 1000 ml graduate until the graduate is filled to the mark. The surface must be even without compressing. Weigh the sample in the graduate to the nearest 0.1 g and calculate the bulk density as follows:

$$\text{Density, bulk (g/ml)} = \frac{\text{wt of sample (g)}}{1000}$$

$$\text{Density, bulk (lbs/cu ft)} = \text{density, bulk (g/ml)} \times 62.435$$

4.4.14 pH

4.4.14.1 Standardization of pH meter. An expanded scale pH meter equipped with a standard calomel electrode and glass electrode shall be used. Standardize the pH meter at pH = 4 at 25°C with a standard buffer solution (e.g. 0.05M potassium hydrogen phthalate) and at pH = 9 at 25°C with a standard buffer solution (e.g. 0.01M sodium borate).

4.4.14.2 Procedure. Dissolve 8g of sample in deionized water and bring up to 100 ml. Determine the pH of the solution at 25°C.

4.4.15 Granulation. Select and weigh the required sieves. If more than one sieve is required, nest the series in increasing order of fineness with the coarsest sieve on top so that material passing through a sieve is transferred directly to the next sieve in the series. Place the sieves on a bottom pan. Weigh 100 g of the sample to the nearest 0.1 g and transfer it to

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the first (coarsest) sieve and cover the assembly. Sift for 4 minutes with a mechanical shaker geared to produce 150 ± 10 taps of the striker per minute. Record the weight of the residue retained on each sieve and calculate the percent passing through or retained on each sieve as follows:

$$\text{Retained, percent} = \frac{A + B \times 100}{W}$$

$$\text{Through, percent} = \frac{W - (A + B)}{W} \times 100$$

Where: A = weight retained on designated sieve, in g
 B = weight retained on sieves nested above designated sieve, in g
 W = weight of sample, in g

4.4.16 Water-vapor transmission rate. The 10 bags selected for this test (see 4.3.3.3) shall be emptied of their contents, without disturbing the seams, and tested in accordance with the provisions of L-P-378.

5. PREPARATION FOR DELIVERY

5.1 Packing, Level C - Ammonium nitrate, prilled, shall be packed in accordance with Code of Federal Regulations, Title 49, Parts 1-199. The net weight of the bag shall be 50 pounds and the type of bag shall be either a multi-wall paper bag or an all-plastic bag. In addition, the bag shall meet the requirements of paragraph 5.1.1 or 5.1.2.

5.1.1 When a paper bag is utilized, the innermost ply shall be a 3 mil thick polyethylene liner and the outermost ply shall be made from wet strength shipping sack kraft paper. The inside liner shall conform to L-P-378, Type II, Class 1; and if a longitudinal seam exists, the seam shall be continuously and securely bonded by fuzing or by a hot melt adhesive. The recommended bag style is a sewn bottom, open mouth, flat tube paper shipping sack and the recommended closures for this bag are the continuous fuzing of the inside polyethylene liner to extend no more than $1/2$ " from the sewn line of bag closure.

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5.1.2 When a plastic bag is utilized, the plastic shall meet the requirements of L-P-378, Type II, Class 1, and both closures shall be fused. The technique of puncturing the bag to vent the trapped air is not permitted but a maze type closure for venting prilled ammonium nitrate may be permitted (see 6.7).

5.2 Marking. Marking shall be in accordance with MIL-STD-129 and shall include but is not limited to the following information:

5.2.1 A yellow label for oxidizing materials shall be applied to or printed on each shipping container.

5.2.2 Each bag of ammonium nitrate shall be marked as follows:

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a b c d e

Where: a = manufacturer's identification
b = first letter of symbol or location of manufacturing plant.
c = lot interfix number for change in process.
d = lot number (sequence numbers) ⁽¹⁾
e = month and year of manufacture

(1) If the ammonium nitrate is manufactured on more than one line, the line should be specified in the lot number as A or B.

6. NOTES

6.1 Ordering data. Procurement documents shall specify the following:

- a. Title, number and date of this specification.
- b. Unit quantities required,
- c. Weight per package unit.
- d. Provisions for submission of first article samples.
- e. Description sheets shall be prepared for each lot in accordance with MIL-STD-1171.

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6.2 Inspection code numbers. The five digit code numbers assigned to the inspections herein are to facilitate future data collection and analysis by the Government. These code numbers are also used to correlate the characteristics cited on Equipment Lists with the inspections listed in this specification. In addition, they should be cited as references on drawings of equipment designs submitted by the contractor to the Government for approval (see 6.3.3).

6.3 Inspection Equipment Designs. Inspection equipment designs are of two types - Government Special Inspection Equipment (SIE) designs and contractor designs. SIE designs are designated by drawing numbers (on the Equipment Lists referenced on the Equipment Tabulation) or (under the "Method of Inspection" headings in Section 4). Design responsibility for all other inspection equipment is assigned to the contractor. Equipment to be designed by the contractor is designated "CD". The contractor need not furnish any design when a complete Government SIE design is provided. Unless otherwise specified, however, the contractor may submit alternate or modified contractor designs of SIE in accordance with 6.3.2 and 6.3.3 should he elect to do so.

6.3.1 SIE designs. SIE designs may consist of any of the following:

- a. Detail drawings which completely depict all information necessary for the fabrication and use of the item of inspection equipment.
- b. A source control drawing or a specification control drawing as defined in MIL-STD-100.
- c. An envelope drawing, as defined in MIL-STD-100, which establishes the criteria which a detail design shall meet. When envelope drawings are specified, the contractor shall prepare designs which comply with the criteria herein.

6.3.2 Contractor designs. Contractor designs are required for all inspection equipment for which SIE designs are not specified and may include commercial equipment which the contractor proposes to use. (Commercial equipment is defined as unmodified equipment which is cataloged and available for purchase by the general public). Contractor

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designs shall include appropriate operating instructions, calibration procedures and maintenance procedures. Commercial equipment shall be fully described by catalog listings or other means which provide sufficient information to permit identification and evaluation by the Government and may include illustrations and engineering data. Designs shall be prepared for any special fixture(s) required to be used with commercial equipment, or with SIE designs if not otherwise covered thereby (see 6.3.1c). Designs shall be of the category and form (per MIL-D-1000) specified in the Contract Data Requirements Lists (DD Form 1423). The contractor is referred to MIL-HDBK-204, "Inspection Equipment Design" for guidance. The specification number and the applicable five-digit defect code number (or other specific identifying information) from Section 4 of this specification shall be referenced on each contractor design together with the component or assembly drawing number and revision letter to which the specific design applies.

6.3.3 Submission of designs for approval. Contractor designs shall be approved by the Government prior to fabricating or procuring the equipment. Designs shall be submitted for approval to Commander, Picatinny Arsenal, Dover, New Jersey 07801, ATTN: SARPA-QA-A-P in accordance with the stipulations, time frame and distribution specified in the Contract Data Requirements List (DD Form 1423) or in the contract. Partial submission of inspection equipment designs is permissible and encouraged. However, the completion date for design review will be based on the date of the final submission of designs. Picatinny Arsenal design review will normally be accomplished within one month after receipt.

6.4 Intended use. The material covered by this specification is intended for use in Minol-2.

6.5 Applicable to the manufacturer of Minol-2 only. Twenty four hours prior to using the ammonium nitrate, select five 25 gram samples from five different bags of ammonium nitrate. These samples should be tested for moisture in accordance with 4.4.1. If any sample fails to comply with the requirements of 3.2, the lot of ammonium nitrate should be dried to meet the requirement.

6.6 One type of prilled ammonium nitrate which has been found satisfactory for the intended purpose

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is made by Mississippi Chemical Corporation, Yazoo City, Mississippi. Similar materials from other sources must be submitted to Picatinny Arsenal, Dover, New Jersey, 07801, ATTN: SARPA-QA-A-P for determination of equivalency to referenced material.

6.7 A suitable type bag with a maze type closure for venting prilled ammonium nitrate is manufactured by Owen-Illinois, Valdosta, Ga. and has been tested and approved by Picatinny Arsenal. Similar bags with maze type closures from other sources must be submitted to Picatinny Arsenal, Dover, New Jersey, 07801, ATTN: SARPA-QA-A-P for determination of either their equivalency to referenced bag or suitability for end item use.

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ARMY-MU

Project Number: 1376-A014

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