MIL-A-23442A(Wep)
3 March 1965
SUPERSEDING
MIL-A-23442(Wep)
16 October 1962

MILITARY SPECIFICATION
AMMONIUM PERCHLORATE

This specification has been approved by the
Bureau of Naval Weapons, Department of the Navy

1. SCOPE

1.1 Scope. This specification covers the requirements for one grade of granular ammonium perchlorate.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

Federal

RR-S-366	Sieves, Standard for Testing Purposes

Military

MIL-P-116	Preservation, Methods of

STANDARDS

Military

MIL-STD-129	Marking for Shipment and Storage

(When requesting any of the above documents, give the title and complete designation of the item shown above. Copies of this specification and other unclassified specifications, standards, and publications required by contractors in connection with specific procurement functions may be obtained from the Commanding Officer, Navy Supply Depot (CDS), 5801 Tabor Avenue, Philadelphia, Pennsylvania, 19120.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the

issue in effect on date of invitation for bids or request for proposal, shall apply.

PUBLICATIONS

Interstate Commerce Commission

49-CFR 71-90	Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D. C., 20102. Orders for the above publication should cite the latest edition and supplements thereto.)

3. REQUIREMENTS

3.1 Preproduction samples. Unless otherwise specified in the contract or order, preproduction samples of the ammonium perchlorate shall be manufactured using the methods and procedures proposed for the production. The sample will be tested as specified in Section 4 herein and is for the purpose of determining that, prior to starting production, the contractor's production methods are capable of yielding items that comply with the technical requirements of the contract. After satisfactorily passing all the preproduction tests specified herein, no changes in raw materials and processing of materials for production shall be made without prior written approval of the procuring activity.

3.2 Data requirements. No data is required by this specification or by applicable documents referenced in Section 2 unless specified in the contract or order (see 6.2).

3.3 Physical and chemical properties

3.3.1 Chemical composition. The chemical composition of the ammonium perchlorate shall be as specified in Table I.

Table I. Ammonium Perchlorate Chemical Composition

Ingredient	Weight Percent
Ammonium perchlorate	99.3 (minimum)
Water insoluble	0.006 (maximum)
Bromates, as $NaBrO_3$	0.004 (maximum)
Chlorides, as NH_4Cl	0.030 (maximum)
Chlorates, as $NaClO_3$	0.020 (maximum)
Chromates, as K_2CrO_4	0.015 (maximum)
Iron, as Fe	0.001 (maximum)
Ash(H_2SO_4 treated)	0.300 (maximum)
Total water	0.050 (maximum)
Total volatiles	0.040 (maximum)

3.3.2 Physical properties. The physical properties of the ammonium perchlorate shall conform to the following:

3.3.2.1 Particle size. The weighted average particle diameter shall be no less than 190 and no greater than 210 microns. Particle size shall also conform to the following requirements:

a. The weight percent of material that shall pass through a Number 50 U.S. Standard Screen shall be no less than 89 and no greater than 97 percent.

b. The weight percent of material that shall pass through a Number 200 U.S. Standard Screen shall be no less than 2 and no greater than 6 percent.

3.3.2.2 Clarity and color. A 10 percent aqueous solution of the ammonium perchlorate shall be no darker than a standard solution of 0.0050 grams of potassium chromate per liter of distilled water.

3.3.2.3 pH value. The pH of a saturated aqueous solution shall be no less than 4.0 nor more than 6.0.

3.3.2.4 Heat stability. The material shall be stable at 177 ± 2 degrees Centigrade (C) for no less than 3 hours.

3.4 Workmanship. The material shall be uniform in quality and shall be free from impurities and other defects that could adversely affect its use.

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, the supplier may utilize his own facilities or any commercial laboratory acceptable to 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.

4.2 Classification of inspections. Inspection of the ammonium perchlorate shall be classified as follows:

a. Preproduction inspection (see 4.4)
b. Quality conformance inspection (see 4.5)

4.3 Sampling

4.3.1 Preproduction sample. A preproduction sample of sufficient quantity of the ammonium perchlorate manufactured in accordance with 3.1 shall be subjected to 3 each of the preproduction tests detailed in 4.4 at an activity designated by the procuring activity. Further production of the ammonium perchlorate by the supplier, prior to the approval of the preproduction sample, shall be at the supplier's risk.

4.3.2 Quality conformance inspection sampling. Unless otherwise specified, sufficient material shall be taken from each lot to perform the tests as specified in 4.5.

4.3.3 Inspection lot. An inspection lot of ammonium perchlorate shall consist of all material presented for acceptance at one time and produced in a single manufacturing run under homogeneous conditions of manufacture.

4.4 Preproduction inspection. The preproduction sample shall satisfactorily pass the quality conformance inspections detailed in 4.5.

4.5 Quality conformance inspection

4.5.1 Visual examination. Visually examine each container in a lot for conformance with 3.4 and Section 5.

4.5.2 Ammonium perchlorate assay. Transfer 5.0 grams (gm) of the dried sample weighed to the nearest milligram (mg) to a glass-stoppered flask containing 40 milliliters (ml) of distilled water and allow to dissolve. (Add a solution of 20 ml of formaldehyde mixed with 20 ml of distilled water, neutralized with 1 normal (N) sodium hydroxide using 3 drops of phenolphthalein indicator.) Mix and allow to stand 30 minutes. Add 2 drops of phenolphthalein indicator and titrate with 1N sodium hydroxide to a pink end point which persists for 5 minutes. Calculate the percent ammonium perchlorate as follows:

$$\text{Percent } NH_4ClO_4 = \frac{11.75\ (A)\ (N)}{W}$$

where:

A = Volume of sodium hydroxide used for titration, ml
N = Normality of sodium hydroxide
W = Weight of sample, gm

4.5.3 Water insolubles. Dissolve a 25.0-gm portion of the sample, weighed to 0.1 gm, in approximately 175 ml of distilled water, warming slightly if necessary. Filter the solution through a dried, weighed, and tared (to nearest 0.1 mg) of filtering crucible. Wash with hot water. Dry to constant weight at 105 ± 5 degrees C, cool in a desiccator and reweigh to the nearest 0.1 mg. Calculate the percent insoluble matter as follows:

$$\text{Percent water insolubles} = \frac{(W_2 - W_1)\ 100}{S}$$

where:

W_1 = Weight of crucible, gm

W_2 = Weight of crucible plus residue, gm

S = Weight of sample, gm

4.5.4 Bromates as sodium bromate. Transfer a 5-gm portion of sample, weighed to 0.1 mg, to a glass-stoppered 500-ml flask, dissolve with approximately 200 ml of cool, freshly boiled, distilled water, and filter. To filtrate add 10 ml of 1N hydrochloric acid, 1 gm of potassium iodide, and 5 ml of 0.2 percent starch solution. Stopper the flask, agitate the solution, and allow to stand in a dark place for one hour. Titrate with 0.02N sodium thiosulfate solution until the blue color disappears. Prepare a blank in the same manner, using similar quantities of reagents. Calculate the percent sodium bromate in the sample as follows:

$$\text{Percent } NaBrO_3 = \frac{1.51\ (A - B)\ (N)}{W}$$

where:

A = Volume of sodium thiosulfate used for sample, ml

B = Volume of sodium thiosulfate used for blank, ml

N = Normality of sodium thiosulfate

W = Weight of sample, gm

4.5.5 Chlorides as ammonium chloride. The chloride content of the material shall be determined by turbidimetric precipitation as silver chloride and comparison with a standard.

a. Preparation of test sample solution. Dissolve a 1.0-gm portion of sample, weighed to 0.1 mg, in approximately 45 ml of water and filter. Transfer filtrate to a Nessler tube. Add 1 ml of concentrated nitric acid, 1 ml of 2-percent silver nitrate solution, dilute to 50 ml, and mix thoroughly.

b. Preparation of comparison standard. Prepare a solution containing 1 ml concentrated nitric acid, 1 ml of 2-percent silver nitrate solution, and water to dilute to 50 ml.

c. Procedure. Add standard chloride solution containing an equivalent of 0.00015-gm ammonium chloride per ml until the turbidity of the comparison sample matches that of the test sample.

Calculate the percent chlorides as follows:

$$\text{Percent } NH_4Cl = \frac{100\ (A)\ (B)}{W}$$

where:

A = Volume of standard chloride solution required to match sample, ml

B = Weight of ammonium chloride per ml of standard solution, gm

W = Weight of sample, gm

Note.- If the sample contains more than 0.03 percent chlorides an additional test shall be performed as follows:

a. Dissolve a 5.0-gm portion of sample, weighed to nearest 0.1 gm, in approximately 45 ml of water and filter. Dilute filtrate to 50 ml and transfer to a 250-ml glass-stoppered flask. Add 10 ml of 5-percent (by volume) nitric acid. Pipet 10 ml of 0.02N silver nitrate solution into the flask. Add 1 ml of nitrobenzene to the flask. Shake vigorously for one minute. Add 10 ml of 5-percent (by weight) ferric nitrate indicator. Back titrate with 0.02N ammonium thiocyanate to a pink color which persists for no less than 30 seconds after vigorous shaking.

b. Prepare a blank and back titrate to the same point.

c. Calculate the percent chlorides as follows:

$$\text{Percent } NH_4Cl = \frac{5.3\ (B - S)\ N}{W}$$

where:

W = Weight of sample, gm

B = Volume of ammonium thiocyanate added to blank solution, ml

S = Volume of ammonium thiocyanate added to sample solution, ml

N = Normality of ammonium thiocyanate

4.5.6 Chlorates as sodium chlorate. Transfer a 1-gm portion of sample, weighed to the nearest 0.1 mg, to a glass-stoppered Erlenmeyer flask. Add, at a temperature of approximately 40 degrees C, 30 ml of boiled distilled water and 40 ml of a solution containing one part by volume concentrated hydrochloric acid to one part water. Heat at approximately 40 degrees C for approximately 30 minutes. Add 1 ml of O-tolidine reagent, made by mixing 1 gm of O-tolidine in one liter of 10 percent hydrochloric acid, and mix. Allow to stand for five minutes at room temperature and compare the color with that of standards prepared from standard potassium chlorate and treated in the same manner as the sample. This determination will include bromate. A correction for sodium bromate shall be included in the calculation as follows:

$$\text{Percent } NaClO_3 = \frac{100\ A}{W} - 0.69\ B$$

where:

A = Weight of sodium chlorate (calculated) equivalent to the weight of potassium chlorate in closest standard, gm

B = Percent sodium bromate in sample (4.5.4)

W = Weight of sample, gm

4.5.7 Chromates as potassium chromate. Dissolve 1 gm of sample, weighed to the nearest 0.1 mg, in approximately 95 ml of water, filter, and dilute filtrate in a Nessler tube to 100.0 ml. Add 3.0 ml of a solution containing one part concentrated sulfuric acid to five parts water and mix. Add 1.0 ml of 0.25-percent diphenyl carbazide solution in alcohol and mix. Prepare a blank in the same manner and add standard (0.0004 gm per ml) potassium chromate solution until the color matches that of the sample

solution. Calculate the percent potassium chromate as follows:

$$\text{Percent } K_2CrO_4 = \frac{0.04\ A}{W}$$

where:

A = Volume of standard potassium chromate solution added to blank, ml

W = Weight of sample, gm

4.5.8 Iron. Transfer 1 gm of sample, weighed to the nearest 0.1 mg, to an evaporating dish. Add approximately 3 ml of distilled water and 2 ml concentrated hydrochloric acid, and evaporate to dryness on steam bath. Dissolve residue in 1 ml of concentrated hydrochloric acid and approximately 25 ml of water. Transfer solution to a Nessler tube and add 3 ml of 25-percent ammonium thiocyanate. Simultaneously prepare a blank in the same manner and add standard (0.00005 gm of iron per ml) iron solution until the pink color matches that of the sample solution. Calculate the percent iron as follows:

$$\text{Percent Fe} = \frac{100\ (A)\ (B)}{W}$$

where:

A = Volume of standard iron solution added to blank, ml

B = Weight of iron per ml of standard solution, gm

W = Weight of sample, gm

4.5.9 Sulfated ash. Ignite a silica crucible with lid at a dull red heat for 30 minutes, cool in a desiccator, and weigh to nearest 0.1 gm. Weigh 5.0 gm of sample to the nearest 0.1 gm. Place the crucible with lid on a hot plate at high heat. Add the sample in small increments and allow to burn completely, covering the crucible after each addition. Add several drops of concentrated sulfuric acid and allow to fume off. When dry, place in a muffle furnace at 600 to 700 degrees C for 30 minutes, cool in a desiccator, and weigh.

Calculate the percent sulfated ash as follows:

$$\text{Percent sulfated ash} = \frac{100\ (W_2 - W_1)}{S}$$

where:

W_2 = Weight of crucible plus residue, gm

W_1 = Weight of crucible, gm

S = Weight of sample, gm

4.5.10 Determination of total water. The water content of the material shall be determined by dissolving the sample in a neutralized methanol solution and titrating indirectly by adding an excess of Karl Fischer reagent and back titrating with a standard water-methanol solution.

4.5.10.1 Materials. The following specialized items shall be used in this determination:

a. Equipment

Aquameter, Model KF-2 with 10-ml burettes, manufactured by Bechman Instruments Inc., or equivalent. Use of an equivalent item of equipment will necessitate use of that manufacturer's particular instructions.

b. Chemicals

Karl Fischer reagent, 3.0 to 5.0 mg water equivalent to one ml

Karl Fischer reagent, standardized to nearest 0.1 mg water per ml.

Methanol, anhydrous, ACS, less than 0.1 percent water.

Water in methanol reagent, 3.0 to 5.0 mg water per ml methanol.

4.5.10.2 Determination of the ratio (R) of Karl Fischer reagent to water-methanol solution. Add an excess of Karl Fischer reagent to approximately 100 ml of methanol and back titrate to the end point. Take burette readings. Allow the solution to stir for 5 minutes. Add 3.0 to 5.0 ml of Karl Fischer reagent and back titrate with water-methanol reagent. Record the volumes used and determine the ratio R (ml Karl Fischer reagent per ml water-methanol reagent). Repeat this determination and obtain an average of the two ratios. The range of the calculated ratios shall be no more than 0.04.

4.5.10.3 Test procedure. Neutralize the moisture in approximately

100 ml of anhydrous methanol by adding an excess of Karl Fischer reagent and back titrating with water-methanol solution to the end point. Record the burette readings. Introduce 5.0 ± 0.1 gm of sample to the neutralized methanol solution. Allow to stir for 5 minutes. Add a visual excess of Karl Fischer reagent. Back titrate with water-methanol to the end point. Record the burette readings and calculate the volumes of Karl Fischer reagent and water-methanol added to the sample solution. Calculate the percent water as follows:

$$\text{Percent water} = \frac{0.1\ F\ (K - MR)}{W}$$

where:

F = Weight of water equivalent to one ml of Karl Fischer reagent, mg/ml

K = Volume of Karl Fischer reagent added to sample solution, ml

M = Volume of water-methanol added to sample solution, ml

R = Ratio of ml Karl Fischer reagent to ml water-methanol

W = Weight of sample, gm

4.5.11 Determination of total volatile matter. Transfer a 10-gm portion of sample, weighed to the nearest 0.1 mg, to a tared wide-form moisture dish and weigh. Place in a gravity-convection oven and allow to remain for 2 hours at 177 ± 2 degrees C. Cool in a desiccator, and weigh. Calculate the percent volatile matter as follows:

$$\text{Percent volatile matter} = \frac{W_1 - W_2\ 100}{S}$$

where:

W_1 = Weight of dish plus sample, gm

W_2 = Weight of dish plus sample after heating, gm

S = Weight of sample, gm

4.5.12 Particle size determination. The weighted average particle size shall be determined by the following procedure:

4.5.12.1 Test procedure:

a. Transfer no less than 500 gm of sample to the riffle sampler

(with 18, 1/2-inch openings discharging in opposite directions). Divide the sample, using the riffle sampler. Discard the material in the right-hand pan. Redivide the material from the left-hand pan, using the riffle sampler. Again discard the material in the right-hand pan.

b. Assemble the sieves in order of mesh size (numbers 50, 70, 100, 140, 200, meeting RR-S-366) with the coarsest sieve on top. Include a bottom pan in the assembly. Transfer 100 gm, weighed to the nearest 0.1 gm, of the material from the left-hand pan of the riffle sample to the top sieve. Add cover plate to the assembly and place in sieve shaker. (Ro-Tap with automatic timer; W. S. Tyler Company or equivalent.) Vibrate for 13 ± 1/2 minutes.

c. Starting with the top sieve, use a brush to transfer the contents of the sieve to a sheet of glazed paper. Weigh the material to the nearest 0.1 gm and record the weight and sieve number. Repeat the transferring, weighing, and recording for each sieve and the bottom pan.

4.5.12.2 Calculations

a. Calculate the weighted average particle size as follows:

$$\bar{D}_W = \frac{(358.5W_{50}) + (253.5W_{70}) + (179.5W_{100}) + (127.0W_{140}) + (89.5W_{200}) + (50.3W_P)}{W_{50} + W_{70} + W_{100} + W_{140} + W_{200} + W_P}$$

where:

$\bar{D}_W$ = Weighted average particle size, microns

W_{50} = Weight of sample retained on 50-mesh screen, gm

W_{70} = Weight of sample retained on 70-mesh screen, gm

W_{100} = Weight of sample retained on 100-mesh screen, gm

W_{140} = Weight of sample retained on 140-mesh screen, gm

W_{200} = Weight of sample retained on 200-mesh screen, gm

W_P = Weight of sample in bottom pan, gm

b. The weight percent of sample passed through the 50-mesh screen shall be calculated as follows:

$$W_{50pass} = \frac{(\text{wt. sample - wt. retained}) \times 100}{\text{weight of sample}}$$

c. The weight percent of sample through the 200-mesh screen shall be calculated as follows:

$$W_{200pass} = \frac{100W_{pan}}{\text{weight of sample}}$$

4.5.13 Clarity and color. Dissolve a 10-gm portion of sample in 100 ml of water and transfer 100 ml of solution to a Nessler tube. Compare with a standard containing 0.005 gm of potassium chromate per liter.

4.5.14 Determination of pH value. In a volumetric flask, prepare 100 ml of saturated solution of sample in freshly boiled and cooled distilled water. Determine the pH of this solution at room temperature using a potentiometric pH meter, (Model 28, Coleman Instruments, Inc., or equivalent) with glass and calomel electrodes.

4.5.15 Heat stability determination. Transfer four 10-gm portions of sample, weighed to 0.1 mg, to 60-millimeter diameter aluminum weighing dishes which have previously been heated to approximately 700 degrees C and cooled. Place on shelf located four inches from top of gravity-convection oven. Maintain oven temperature at 177 ± 2 degrees C. Remove one sample from the oven after 2 hours and an additional sample every hour thereafter. Cool the sample in a desiccator 20 to 30 minutes. Add approximately 5 gm of cooled sample to a mixture of 100 ml distilled water, 0.5 gm granular potassium iodide, and 5 ml of 0.2-percent starch solution. Stir to dissolve ammonium perchlorate. The solution shall remain almost colorless. Immediate appearance of violet or blue color indicates decomposition. Repeat the procedure for each sample removed from the oven.

4.6 Acceptance criteria

4.6.1 Preproduction. Failure of any sample to meet any requirement of this specification shall be cause for rejection of the lot.

4.6.2 Quality conformance. Failure of any sample to meet any requirement of this specification shall cause rejection of the lot.

5. PREPARATION FOR DELIVERY

5.1 Preservation, packaging, and packing. Unless otherwise specified by the procuring activity, the preservation, packaging, and packing shall be in accordance with the Code of Federal Regulations 49-CFR 71-90, and MIL-P-116, method II, optional submethod.

5.2 Marking. Each container shall be marked in accordance with MIL-STD-129. Marking shall include, but not be limited to, the following information:

a. Manufacturer's name and location
b. Material trade name
c. Net weight or volume
d. Lot number, batch number, and date of manufacture
e. Shelf life or storage limitations
f. Number and revision letter of this specification

6. NOTES

6.1 Intended use. The material purchased in accordance with this specification is intended to be used as an ingredient in some types of rocket motor fuels.

6.2 Ordering data. Procurement documents should specify, but not be limited to, the following information:

a. Title, number, and revision letter of this specification
b. Minimum lot size, if applicable
c. Whether preproduction sample is required
d. Place of delivery
e. Size of container
f. Request for test data
g. Type and size of container

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 119-R004

INSTRUCTIONS

This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity *(as indicated on reverse hereof)*.

SPECIFICATION
MIL-A-23442A(WP) AMMONIUM PERCHLORATE

ORGANIZATION *(Of submitter)*

CITY AND STATE

CONTRACT NO.

QUANTITY OF ITEMS PROCURED

DOLLAR AMOUNT
$

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ SUBCONTRACT

1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?

A. GIVE PARAGRAPH NUMBER AND WORDING.

B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES.

2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID

3. IS THE SPECIFICATION RESTRICTIVE?

☐ YES ☐ NO IF "YES", IN WHAT WAY?

4. REMARKS *(Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)*

SUBMITTED BY *(Printed or typed name and activity)*

DATE

DD FORM 1 APR 63 1426

C-8279

FOLD

DEPARTMENT OF THE NAVY
Bureau of Naval Weapons
Washington, D. C. 20360

POSTAGE AND FEES PAID
NAVY DEPARTMENT

OFFICIAL BUSINESS

BUREAU OF NAVAL WEAPONS (RREN-5)
DEPARTMENT OF THE NAVY
WASHINGTON, D. C. 20360

FOLD