MILITARY SPECIFICATION

AMMONIUM PERCHLORATE FOR SOLID PROPELLANT GRAINS MARK 75 AND MARK 76

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

1. SCOPE

1.1 This specification establishes the minimum requirements for a special grade ammonium perchlorate for the solid propellant grains Mark 75 and Mark 76.

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

Military

MIL-D-3464	Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification

Federal

RR-S-366	Sieves, Standard for Testing Purposes

STANDARDS

Military

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129	Marking for Shipment and Storage

(When requesting any of the applicable documents, refer to both title and number. All requests should be made via the cognizant Government Inspector. Copies of this specification and other unclassified specifications and drawings required by contractors in connection with specific procurement functions should be obtained upon application to the Commanding Officer, Naval Supply Depot (Code 105), 5801 Tabor Avenue, Philadelphia 20, Pennsylvania. All other documents should be obtained from the procuring activity or as directed by the contracting officer.)

3. REQUIREMENTS

3.1 Preproduction. - The preproduction test sample shall be manufactured using the process methods and equipment proposed for production. Any significant changes after preproduction approval must be approved by the procuring activity prior to incorporation into production. It shall be the responsibility of the contractor to inform the procuring activity of proposed changes after approval of the preproduction sample. In the event of significant changes in the process methods and equipment which in the opinion of the procuring activity may adversely affect the characteristics of the material, additional preproduction samples may be required.

3.2 Chemical Properties. - The chemical properties of the ammonium perchlorate shall be in accordance with Table I.

TABLE I

CHEMICAL PROPERTIES

Characteristic	Requirement	
	Minimum	Maximum
Purity,%	99.0	...
Chloride (as NH_4Cl), %	...	0.10
Chlorate (as NH_4ClO_3), %	...	0.02
pH	4.3	5.8
Moisture, %		
Total	...	0.07
Surface	...	0.015
Sulfated Ash, %	...	0.7
Ether Soluble, %	...	0.01
Water Insoluble %	...	0.04
Bromate (as NH_4BrO_3), %	...	0.004

3.3 Physical Properties. - The physical properties of the ammonium perchlorate shall be in accordance with Table II.

TABLE II

PHYSICAL PROPERTIES

Characteristic	Mesh	Requirements	
		Pass	Retained
Granulation (U. S. Standard Sieves)	20	99.9% Min.	
	100		70% Min.
Color		White	
Friability		10% Max.	

3.4 Workmanship. - The ammonium perchlorate shall be a uniform product free from foreign materials. It shall be uniform in quality and manufactured in accordance with standard manufacturing procedures of the industry.

3.5 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).

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 inspection are deemed necessary to assure supplies and services conform to perscribed requirements.

4.2 Classification of Examiniations and Tests. - Examinations and tests of the ammonium perchlorate shall be classified as follows:

(a) Preproduction Tests (4.4)

(b) Quality Conformance Inspection (4.5)

4.3 Lot Size. - A lot shall consist of material produced by one manufacturer in one continuous operation employing not more than one lot of each ingredient and with no change in formulation or process. If manufacture is by batch process, each batch shall constitute a lot.

4.4 Preproduction Tests. - Preproduction tests shall consist of all of the Quality Conformance Inspections and requirements of this specification.

4.5 Quality Conformance Inspection. - The following procedures shall be performed in duplicate for compliance with section 3.

4.5.1 Sampling. - Sampling for quality conformance inspections shall be in accordance with Standard MIL-STD-105, Inspection Level I, except that the sample size shall never be less than three containers. One pound of material shall be considered as one unit of product (but only for purposes of determining the sample size in applying the above Standard). After the sample size has been determined (based on the total number of units of product in the lot), containers equal in number to the sample size shall be selected at random from the lot.

4.5.2 Preparation of Reagents. - The preparation of reagents required for the determination of chlorides, chlorates, and perchlorates shall be as follows:

4.5.2.1 Silver Nitrate .1 Normal. - Weigh out approximately 34 grams (gms) of reagent grade silver nitrate and dissolve in a 2 liter volumetric flask. Dilute to the mark with distilled water. The flask should be wrapped with aluminum foil to keep light out. The reagent is standardized by weighing 0.1500 ± .0010 gms of oven-dried (350°F for 3 hours) potassium chloride and dissolving in a 250 milliliter (ml) beaker containing 50 ml of distilled water. Add to the solution approximately 0.5 gms of potassium chromate and titrate with silver nitrate to light brown end point. The normality of the silver nitrate shall be calucated as follows:

$$N_{AgNO_3} = \frac{\text{wt KCl (gms)}}{0.0745 \times \text{ml of } AgNO_3}$$

4.5.2.2 Potassium Thiocyanate .1 Normal.- Dissolve approximately 18 grams of KCNS and dilute to 2 liters with distilled water in a 2 liter volumetric flask. Standardize the solution by titrating it against 20.00 ± .01(A) ml of standard $AgNO_3$ solution in a 250 ml beaker containing 50 ml of distilled water, add 10 ml of ferric ammonium sulfate indicator solution and 2 ml of nitrobenzene. The end point is determined by development of a faint brown color. The normality of the potassium thiocyanate shall be calculated as follows:

$$N_{KCNS} = \frac{A \times N_{AgNO_3}}{\text{ml KCNS}}$$

4.5.2.3 1-1 Nitric Acid Solution. - Dilute 125 ml of concentrated nitric acid to 250 ml with distilled water in a 250 ml volumetric flask.

4.5.2.4 Ferric Ammonium Sulfate Indicator. - Dissolve 28.0 grams of ferric ammonium sulfate crystals in 80 ml of hot water, cool, filter, and dilute to 100 ml

with nitric acid solution (1-1).

4.5.2.5 Ferrous Sulfate ($FeSO_4.7H_2O$. -Dissolve 176.5 gms of ferrous sulfate in 400 ml of distilled water, in a 1 liter volumetric flask. Add 50 ml of 1-1 sulfuric acid with usual precautions. Solution is cooled and diluted up to 1 liter mark with distilled water.

4.5.3 Determination of Composition. -The compositional characteristics shall be determined as follows:

4.5.3.1 Determination of Percent Ammonium Chloride NH_4Cl. -Weigh out about 10 grams of the ammonium perchlorate sample to the nearest 0.1 milligram (mg) and dissolve in a 250 ml beaker containing 50 ml of distilled water. During vigorous magnetic stirring, add approximately 20.00 ml of $AgNO_3$ solution. Next add 5 ml ferric ammonium sulfate indicator solution and 2 ml of nitrobenzene. Titrate this solution with the standard KCNS solution to a faint brown end point. The percent ammonium chloride shall be calculated as follows:

$$\% NH_4Cl = \frac{(5.35)(a)(A) - (5.35)(b)(B)}{W}$$

Where: A = total ml of $AgNO_3$ solution used
B = total ml of KCNS solution used
a = normality of $AgNO_3$
b = normality of KCNS
W = weight of sample

4.5.3.2 Determination of Percent Chloride (Cl). -The percent chloride (Cl) due to ammonium chloride shall be calculated from the data of 4.5.3.1 as follows:

$$\% Cl = \frac{(aA - bB)}{W} \times 3.545$$

4.5.3.3 Determination of Percent Ammonium Chlorate (NH_4ClO_3). -Weigh out 4 to 5 gms of sample to the nearest 0.1 mg and dissolve in approximately 100 ml of distilled water in a 600 ml beaker. Add to this solution 50 ml of ferrous sulfate solution, cover the beaker with a watch glass, and slowly boil the solution for 15 minutes. Remove from heat, place solution under the lab hood, and slowly add 10 ml of concentratec nitric acid. The solution is then heated slowly to boiling point for 5 minutes, cooled, and then approximately 20.00 ml of $AgNO_3$ solution added. Add 5 ml of ferric alum indicator solution and 2 ml of nitrobenzene, then titrate with KCNS to faint brown end point. The percent ammonium chlorate shall be calculated as follows:

$$\% NH_4ClO_3 = \frac{(aA-bB)\ 3.545}{W} - C\ \frac{(100)}{(34.93)}$$

A = total ml of $AgNO_3$
B = total ml of KCNS
a = normality of $AgNO_3$
b = normality of KCNS
W = weight sample
C = percent Cl due to NH_4Cl

4.5.3.4 Determination of Percent Ammonium Perchlorate (NH_4ClO_4). Weigh out approximately 0.5 gms to the nearest 0.1 mg of sample into a platinum crucible. Add approximately 5 gms of sodium carbonate and stir the materials together using a pyrex stirring rod and then add a layer of sodium carbonate over the surface of the sample. Fuse the sodium carbonate and ammonium perchlorate carefully over a low flame for approximately 30 minutes and then gradually increase the flame to full, until melted.

Place the cooled crucible, containing the fused sample, on its side, in a 600 ml beaker containing 50 ml of 1-1 nitric acid and 100 ml of water. Immediately cover the beaker with a cover glass to prevent splattering outside the beaker. After the fused sample dissolves, remove the crucible after flushing with distilled water. Introduce a magnetic stirring bar and then add 50.00 ml $AgNO_3$. Next add 5 ml of ferric ammonium sulfate indicator and 2 ml of nitrobenzene. The solution is ready for titration with KCNS to light brown end point. The percent ammonium perchlorate shall be calculated as follows:

$$\% NH_4ClO_4 = \frac{11.75\ (aA-bB)}{W} - 2.20C - 1.16D - 0.96E$$

C = percent ammonium chloride NH_4Cl
D = percent ammonium chlorate NH_4ClO_3
E = percent sodium perchlorate $NaClO_4$
A = ml of $AgNO_3$
a = normality of $AgNO_3$
B = ml of KCNS
b = normality of KCNS
W = weight of sample

4.5.3.5 Determination of pH. -Add 50 ml of freshly boiled distilled water, adjusted to pH 7, to 20 gms of composite sample. Stir the solution well, and cool to room temperature. Measure the pH to the nearest 0.01 unit with any standard electrometric equipment.

4.5.3.6 Determination of Moisture Content.

4.5.3.6.1 Total Moisture. -Neutralize the water in a 100 to 125 ml portion of anhydrous methanol (as in the standardization of the reagents), and introduce a sample about 5 gms, weighed to the nearest 0.1 mg, of perchlorate from a stoppered weighing bottle. Determine the weight of the sample from the weight of the bottle with and without the sample. Run in a visible excess of Karl Fischer reagent, and back-titrate with water-methanol solution.

4.5.3.6.2 Surface Moisture. -The surface moisture shall be determined by any conventional laboratory method.

4.5.3.7 Sulfated Ash Determination. -The sulfated ash shall be weighed as solium sulfate (Na_2SO_4) but calculated as sodium perchlorate ($NaClO_4$) using the following procedures:

4.5.3.7.1 Reagent. -The reagent shall be sulfuric acid, approximately 50%. Grind at least 5 grams of dried sample to a fine powder and weigh to the nearest 0.1 mg. Add about 0.2 grams of sample to a previously ignited and tared silica crucible. Cover, and heat carefully with a Bunsen flame until the portion has decomposed. Continue the ignition of small increments until the entire sample has been decomposed and the volatile salts driven off. Allow the crucible to cool, and add 3 ml concentrated H_2SO_4. Heat again, and complete the volatilization of H_2SO_4 at a dull red heat. Cool in a desiccator, and weigh. Calculation of the percent sulfated ash shall be as follows:

$$\% \text{ sulfated ash as } NaClO_4 = \frac{\text{weight of residue} \times 122.5 \times 100}{\text{weight of sample} \times 71}$$

$$= \frac{\text{wt. of residue} \times 172}{\text{wt. of sample}}$$

4.5.3.8 Ether Soluble Organics Determination. -The ether soluble organics determination shall be as follows:

(a) Weigh to the nearest 0.1 mg, a 25.0 ± 0.1 gm sample into extraction thimble of Soxhlet extraction apparatus.

(b) Extract with 100 ml anhydrous ethyl ether for two hours.

(c) Transfer ether extract to tared Vycor dish and evaporate to dryness on water bath.

(d) Run a blank determination on 100 ml ether from same source as (b) above.

(e) Increase in weight minus blank is calculated as organic material.

4.5.3.9 Water Insoluble Determination. -The water insolubles shall be determined as follows:

(a) Dissolve 25.0 g of the composite sample weighed to nearest 0.1 mg in about 250 ml of distilled water, and filter the solution through a tared Selas crucible of medium porosity. Wash with distilled water, dry for 1-1/2 hours at 105°C, cool, and weigh to nearest 0.1 mg.

(b) Calculation:

$$\% \text{ water insoluble} = \frac{\text{weight of residue}}{\text{weight of sample}} \times 100$$

4.5.3.10 Bromate Determination. -The bromate determination shall be as follows:

(a) Reagents: Potassium iodide, analytical reagent; hydrochloric acid, dilute 1:9; starch indicator solution, 0.2; standard $Na_2S_2O_3$ solution, 0.02N.

(b) Weigh 100 grams of sample to the nearest 0.1 mg, into a 500 ml glass stoppered Erlenmeyer flask, and add 200 ml of freshly boiled and cooled distilled water, warm to dissolve if necessary. Add 0.5 g of potassium iodide, 5 ml of 1:9 HCl, and 5 ml of starch indicator solution. Mix well and allow to stand in a dark place for 1 hour. Titrate with standard sodium thiosulfate solution until the blue color disappears. Run a blank on the reagents.

(c) Calculation:

$$\% \text{ bromate as } NH_4BrO_3 = \frac{0.0243\ N\ (V-v) \times 100}{0.1\ S} = \frac{2.43\ N\ (V-v)}{S}$$

Where: V = ml sodium thiosulfate required by sample
v = ml sodium thiosulfate required by blank
N = normality of sodium thiosulfate solution
S = sample weight

4.5.3.11 Granulation Determination.

4.5.3.11.1 Reagent. -The reagent shall be tri-calcium phosphate, Monsanto Chemical Company or approved equivalent.

4.5.3.11.2 Apparatus. -Tyler "RoTap" sieve shaker, W. S. Tyler Company, Cleveland, Ohio, or approved equivalent; U.S. Standard sieves, 8 inch diameter, size numbers 20 and 100 (Specification RR-S-366).

4.5.3.11.3 Procedure. -The procedures for the granulation determination shall be as follows:

(a) Blend the composite sample carefully in the sample container, and weigh out 100.0 g. Add 1.0 g of TCP conditioner, and mix thoroughly by shaking for one minute in a one-pint glass jar.

(b) Assemble the sieves in numerical order from top to bottom, with a pan under the stack, and transfer the mixture to the top sieve. Add the cover to the top of the stack, and place the set in the shaker. Set the timer to run 13 minutes, and start the shaker.

(c) When the shaker has stopped, remove the stack of sieves. Starting with the top sieve, brush any material adhering to the bottom of the sieve into the next lower sieve, and transfer the contents to a sheet of glazed paper. Use a brush to remove any material that remains within the sieve. Weigh the retained material to the nearest 0.1 g.

(d) Clean the material from the remaining sieve, adding the fraction to the preceding one. Record the cumulative weight, which is equal to the percentage cumulative retention of the original sample neglecting the conditioner, which is assumed to pass to the pan).

4.5.3.12 Color Determination. -Determination of color shall be visual.

4.5.3.13 Friability. -The friability shall be determined as follows:

(a) Place 100 gms of ammonium perchlorate on a No. 100 U. S. Standard stainless steel sieve with stainless steel pan and cover lid.

(b) Place assembly on Combs Gyrating Sifting Machine or equivalent and shake for 30 minutes.

(c) Weigh out 50.0 gms of perchlorate remaining on the No. 100 sieve onto a No. 100 U. S. Standard stainless steel sieve with 100 ea. 3 millimeter glass

beads, assemble with cover lid and pan.

(d) Place assembly on the Combs Sifting Machine, attach clappers on each side of the No. 100 sieve and shake for 30 ± 1 minute.

(e) Weigh amount of ammonium perchlorate passing the No. 100 sieve.

(f) Calculate friability $= \frac{\text{Weight passing}}{\text{Initial weight}} \times 100$

4.6 Acceptance Criteria. -All tests results shall indicate compliance with the requirements of section 3. Failure to meet these requirements shall be cause for rejection of the lot.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging. -Preservation and packaging shall be level C or as specified (see 6.2).

5.1.1 Level C. -The ammonium perchlorate shall be packaged in a sealed polyethylene liner with a minimum of one eight-unit bag of MIL-D-3464 desiccant per 50 pounds of ammonium perchlorate. If more than one eight-unit bag of desiccant is used, the bags shall be tied together.

5.2 Packing. -Packing shall be level C or as specified (see 6.2).

5.2.1 Level C. -The product packaged as specified in 5.1 shall be packed in dry moisture proof sealed containers and in a manner to insure carrier acceptance and safe delivery at destination. Containers shall be in accordance with Uniform Freight Classification Rules or regulations of other carriers applicable to the mode of transportation.

5.3 Marking. -Unless otherwise specified by the contract or order, unit packages, intermediate packages, and shipping containers shall be marked in accordance with the requirements of MIL-STD-129 and shall include but not be limited to the followint (see 6.2):

(a) Manufacturer's name and address

(b) Gross and net weights

(c) Lot number and date of manufacture

(d) The number of this specification

(e) Safety precautions or unusual storage requirements

(f) Purchase order or contract number

6. NOTES

6.1 Intended Use. -Material purchased in accordance with this specification is intended for use as an ingredient in solid propellant for Rocket Motors Mark 38 and Mark 39.

6.2 Ordering Data. -Procurement documents shall specify but not be limited to the following information:

(a) Title, number, and date of this specification.

(b) Type and size of container desired.

(c) Place of inspection

(d) Minimum lot size, if applicable.

(e) Place of delivery

(f) Request for copies of inspection data, if source inspected.

(g) Selection of applicable levels of packing and packaging (see 5.1 and 5.2)

(h) Marking requirements (see 5.3)

(i) Data requirements

6.3 Conflicting Requirements. -Conflicting requirements arising between this specification or any specifications, publications, or drawings listed herein shall be referred in writing to the procuring activity or appointed agent for interpretation and clarification.

6.4 Request for Deviation. -Request for deviation from this specification, applicable drawings, specifications, or materials or processes shall be forwarded to the procuring activity prior to incorporating the desired change into production. All

deviations shall be limited to the contract under which they were granted. A deviation is a before-the-fact request.

6.5 Request for Waiver. -Request for waiver from this specification, applicable drawings, specifications, or materials shall be forwarded to the procuring activity. A waiver is an after-the-fact request. A waiver request shall include the following:

(a) Exact nature of the defect.

(b) CD number and defect classification, if any.

(c) Quantity of items involved.

6.6 Value Engineering. -Manufacturers of material covered by this specification are encouraged to submit value engineering suggestions to the procuring activity in an effort to reduce cost.

6.7 Safety Precautions. -This is potentially a dangerous material. All detailed safety precautions of the manufacturing, handling, or using activity shall be strictly observed.

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-23946(Wep) AMMONIUM PERCHLORATE FOR SOLID PROPELLANT GRAINS MK 75 & MK 76

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

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