

MIL-A-82667A(OS)
 22 September 1978
 Superseding
 MIL-A-82667(OS)
 31 January 1977

MILITARY SPECIFICATION

AMMONIUM PERCHLORATE, GENERAL SPECIFICATION FOR

This specification is approved for use by the Naval Sea Systems Command, Department of the Navy, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers the general requirements for ammonium perchlorate.

1.2 Classification. Ammonium perchlorate shall be supplied with or without tricalcium phosphate (TCP) as specified in the applicable detail specification. Purity and granulation shall be as specified in the applicable detail specification.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

* MILITARY

MIL-D-3464	Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification
MIL-P-82646	Plastic Film, Conductive, Heat-Sealable, Flexible
MIL-A-82667/1	Ammonium Perchlorate, High Purity
MIL-A-82667/2	Ammonium Perchlorate, Conditioned

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Ordnance Station, Standardization Division (611), Indian Head, MD 20640, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 6810

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STANDARDS

* FEDERAL

FED-STD-313 Material Safety Data Sheets, Preparation and the Submission of

MILITARY

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

MIL-STD-129 Marking for Shipment and Storage

MIL-STD-414 Sampling Procedures and Tables for Inspection by Variables for Percent Defective

MIL-STD-1218 ACS Chemicals

MIL-STD-1234 Pyrotechnics, Sampling, Inspection and Testing

* PUBLICATIONS

NAVAL SUPPLY SYSTEMS COMMAND

Publication 505/MCO P4030.19 Packaging and Handling of Dangerous Materials for Transporting by Military Aircraft

(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).

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.

AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM B 330-65 Average Particle Size of Refractory Metals and Compounds by the Fisher Sub-sieve Sizer

ASTM D 1348-61 Moisture in Cellulose

ASTM E 11-70 Wire-Cloth Sieves for Testing
Purposes

ASTM E 203-75 Water Using Karl Fischer Reagent

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

CODE OF FEDERAL REGULATIONS

46 CFR 140-149 Shipping

49 CFR 100-199 Transportation

(The Interstate Commerce Commission Regulations are now a part of the Code of Federal Regulations, available from the Superintendent of Documents, Government Printing Office, Washington, DC 20402. Orders should cite "46 CFR 140-149 or 49 CFR 100-199" the latest issue and supplements thereto).

NATIONAL MOTOR FREIGHT TRAFFIC ASSOCIATION, INC AGENT

National Motor Freight Classification

(Application for copies should be addressed to American Trucking Associations, Attn: Traffic Department, 1616 F Street, Washington, D.C. 20036).

UNIFORM CLASSIFICATION COMMITTEE, AGENT

Uniform Freight Classification

(Application for copies should be addressed to the Uniform Classification Committee, Room 1106, 222 South Riverside Plaza, Chicago, IL 60606).

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies).

3. REQUIREMENTS

3.1 Detail specifications. The individual material requirements shall be as specified herein and in accordance with the applicable detail specifications. In the event of any conflict between requirements of this specification and the detail specification, the latter shall govern.

3.2 Chemical and physical requirements. The chemical and physical requirements shall be drawn from the properties in TABLE I. The detail specification shall delineate the specific requirements.

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3.3 Granulation and form. The average particle size, the size distribution and the form of the crystals shall be as given in the detail specification.

3.4 Material safety data sheets. The contractor shall prepare and submit material safety data sheets in accordance with FED-STD-313 as specified in the contract (see 6.4).

3.5 Workmanship. The material shall be uniform, free from contaminants, foreign material or any other defect that would prevent its use for the purpose intended.

TABLE I. Chemical and physical properties.

Composition requirements (wt%)

Purity

Chloride, as NH_4Cl Chlorate, as NH_4ClO_3 Bromate, as NH_4BrO_3 Chromate, as K_2CrO_4 Sulfate, as $(\text{NH}_4)_2\text{SO}_4$

Sulfated ash, calculated

as NaClO_4

Sulfated ash, calculated

as Na_2SO_4 Iron, as Fe_2O_3

Sodium and potassium

Nonalkali metals as oxides

Tricalcium phosphate, as

 $\text{Ca}_3(\text{PO}_4)_2$

Moisture, surface

Moisture, total

Water insolubles

Ether soluble

Total volatiles

Stability (hr)

pH

Friability, %passing

No. 100 (150 μm) sieve

Color

Granulation

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified

in the contract, the contractor 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 that supplies and services conform to prescribed requirements.

4.2 Lot. A lot shall consist of the total quantity of cross-blended material. For non-cross-blended material, a lot shall consist of the quantity produced in a single-batch; or when manufactured by a continuous process, a lot shall consist of the total quantity to be offered for acceptance at one time.

4.3 Sampling. The number of containers sampled shall be in accordance with MIL-STD-414, Level IV. The containers sampled shall be selected by a random process. From each container to be sampled, a primary sample of approximately one (1) pint is taken by means of a grain thief or by another appropriate device inserted to the maximum depth at random locations in the container. Seal each primary sample in a one (1) pint glass jar and label each jar with the name, lot number and container number. The primary samples from a lot shall be grouped and blended to form three composite samples. Each composite sample shall consist of an approximately equal amount of each primary sample from the specified number of primary samples. When a lot consists of less than three containers each container shall be sampled. The sample portions thus obtained shall be grouped and blended to form three composite samples as follows:

If one container is received, three (3) samples are taken, each being treated as a composite sample.

If two containers are received, two (2) samples are taken from one container, one sample is taken from the other container, and each is treated as a composite sample.

Blend each composite thoroughly and label with the name, date, lot number and container numbers. All acceptance tests shall be made on each of the composite samples representing the lot. The average of the test values obtained on the composites shall be used for acceptance purposes. However, if during the sampling or testing it becomes apparent that the lot is not uniform throughout, the inspector may test any of the primary samples for compliance with the specification. Primary samples shall be held for possible future examination, should the composite samples fail to meet the requirements.

4.4 Acceptance tests. The following acceptance tests as well as any additional test listed in the detail specification shall be performed on each lot. The test results shall be compared with the detail specification requirements. Any deviation from the specified value shall result in rejection of the lot. When specified in the contract (see 6.2), the supplier shall submit a report giving the results obtained for all inspections performed and a certified statement that the lot meets all the requirements of this specification. Unless otherwise specified, all

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chemicals shall be ACS grade in accordance with MIL-STD-1218.

4.4.1 Moisture. The Karl Fischer (KF) method shall be used with any suitable apparatus. The general procedure for total moisture content shall be by titration of a solution of the sample in a 3:1 mixture of pyridine and methanol. The surface moisture is determined by titration of a slurry of the solid sample in a solution or liquid medium saturated with ammonium perchlorate. The end point may be determined either visually or electrometrically by means of either a direct or back titration of a measured excess of KF reagent. The direct titration shall be in accordance with ASTM E 203-75 and the back titration shall be in accordance with ASTM D 1348-61. The number of replications, the specimen weight, the water equivalent of the KF reagent and the precision of the determination shall be in accordance with the detail specification. The standardization of the KF reagent shall be performed daily in accordance with ASTM E 203-75. All the standard precautions must be taken to protect the specimens against change in moisture content and to protect all reagents, solvents and the titration mixture against the entry of water. During all the titrations, the stirring action shall be at the highest speed which will not cause splashing or bubble formation.

4.4.2 Purity. The purity of the ammonium perchlorate shall be determined by fusion of the sample with sodium carbonate followed by dissolution in nitric acid, adjustment of the pH with sodium bicarbonate and determination of chloride ion by a Mohr or a Volhard titration with silver nitrate. A blank titration shall be conducted on the same volumes of solution and quantities of reagents as those used in the determination.

Alternatively the perchlorate may be determined by a direct electro-metric titration of chloride ion after sodium carbonate fusion and dissolution in nitric acid. In the calculation of perchlorate, the equivalent of chloride ion corrections shall be applied for chloride and chlorate percentages as impurities in the ammonium perchlorate. A correction for sodium perchlorate may be made if required by the detail specification.

4.4.3 Chloride. The chloride content shall be calculated as ammonium chloride after a determination by a turbidimetric method, a Volhard or a Mohr titration with silver nitrate. Alternatively, chloride may be determined by a direct potentiometric titration. A blank titration shall be conducted on the same volumes of solution and quantities of reagents as those used in the determination.

4.4.4 Chlorate. The chlorate content shall be calculated as ammonium chlorate and shall be determined by reduction with ferrous sulfate solution followed by a Mohr titration with silver nitrate. A blank titration shall be conducted on the same volumes of solution and quantities of reagents as those used in the determination. Alternatively, the chlorate shall be determined colorimetrically in acid solution using o-tolidine reagent by comparison with a standard chlorate solution.

4.4.5 Bromate. The bromate content calculated as ammonium bromate shall be determined by oxidation of iodide to free iodine followed by a thiosulfate titration. A blank titration shall be conducted on the same volumes of solution and quantities of reagents as those used in the determination.

4.4.6 Sulfated ash. The sulfated ash weighed as sodium sulfate and calculated as sodium perchlorate shall be determined by sulfuric acid digestion of the sample followed by volatilization of volatile salts and weighing the residue.

4.4.7 Iron. The iron content of the sample, calculated as ferric oxide shall be determined on the residue from the sulfated ash determination. The residue shall be dissolved in concentrated hydrochloric acid and diluted in a volumetric flask. The resulting solution shall be compared colorimetrically with a standard solution with a known iron content using either potassium permanganate and thiocyanate or hydroxylamine and o-phenanthroline for the colorimetric end point.

4.4.8 pH. The pH shall be determined on a saturated solution of the sample dissolved in hot distilled water and reprecipitated using a suitable potentiometer having glass-calomel electrodes.

4.4.9 Sodium and potassium. The sodium and potassium content shall be determined by flame photometry.

4.4.10 Phosphate. The phosphate content of the sample shall be determined by comparing the absorbance of the sample solution with a calibration curve of concentration of a vanadate molybdate complex versus absorbance.

4.4.11 Water insolubles. A sample of the ammonium perchlorate shall be dissolved in distilled water. The solution shall be filtered and the residue on the filter weighed.

4.4.12 Granulation. The average particle size and the particle size distribution shall be determined by one of the following four methods in accordance with the detailed specification.

a. Particle size shall be determined in accordance with ASTM B 330-65.

b. Particle size shall be determined in accordance with MIL-STD-1234, Method 201.1.

c. Particle size shall be determined using a stack of eight-inch (8") diameter U.S. Standard sieves conforming to ASTM E 11-70 shaking with a Ro-Top shaker and timing with an automatic timer. The results shall be reported as cumulative weight percent retained on each sieve.

d. The same procedure of c. above shall be used and the results calculated as a cumulative weight percent and a weighted average mean diameter.

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4.4.13 Visual inspection. All samples shall be visually inspected to verify conformance to the workmanship requirements.

4.4.14 Packaging inspection. The packaging, packing and marking shall be inspected to verify conformance with the requirements of section 5. Sampling shall be in accordance with MIL-STD-105 with an AQL of 2.5%.

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5. PACKAGING

5.1 Preservation. Preservation shall be level A.

5.1.1 Level A. Level A preservation shall consist of desiccant conforming to Type I of MIL-D-3464 included in each container as specified in the contract (see 6.2).

5.2 Packaging. Packaging shall be level A or level C as specified in the detail specification (see 6.2).

5.2.1 Level A. A maximum of 100 lbs of ammonium perchlorate that consists of 50% by weight or more of 15 μ m or smaller particles shall be packaged in a DOT specification 37A steel drum in accordance with 49 CFR 178.131. The drum shall be lined with a conductive plastic liner in accordance with MIL-P-82646 and shall have a maximum capacity of 30 gallons. Shipment shall be by motor vehicle or rail freight. Trailer on flat car service shall not be authorized.

5.2.2 Level C. A maximum of 100 lbs of ammonium perchlorate that consists of less than 50% by weight of 15 μ m or smaller particles shall be packaged in accordance with 49 CFR 171.153, 171.154 and 173.239(a) or in containers of equal or greater strength and efficiency that have been approved by the procuring activity.

5.3 Marking. Marking shall be level A or level C as specified in the detail specification (see 6.2).

5.3.1 Level A. Level A packages shall be marked in accordance with MIL-STD-129, 49 CFR 171-178, and the following:

a. Container Markings:

"HIGH EXPLOSIVES - DANGEROUS (AMMONIUM PERCHLORATE) -
(Letters shall be at least 7/16 inch in height)
U.S. COAST GUARD IX-E
DOT SPECIAL PERMIT NO. 4041 APPLIES"

Title, number and date of this specification
Manufacturer's name and location
Type and class of material
Net weight
Lot number, batch number(s), and date of manufacture
Storage conditions
Contract number

b. Bill of Lading Description:

"High Explosives (Ammonium Perchlorate, NOIBN, Class A)
 UFC #11, Item 22430, NMFC 13, Item 43050
 DOT Special Permit No. 4041 applies"

c. Label requirement: Orange label (per 49 CFR 173.405) for air shipments apply "Explosive A" Label as required by Rule No. 5 of NAVSUPPUB 505/MCO P4030.19

d. U. S. Coast Guard Classification: CLASS IX-B

e. Quantity Distance: Group 7

f. Fire Fighting Specification (DD Form 836): Group V

5.3.2 Level C. Level C packages shall be marked in accordance with MIL-STD-129, 49 CFR 171-178 and the following:

a. Container Marking:

"Ammonium Perchlorate, Oxidizing Material - Keep Fire Away"
 Title, number and date of this specification
 Manufacturer's name and location
 Type and class of material
 Net weight
 Lot number, batch number(s), and date of manufacture
 Storage conditions
 Contract number

b. Bill of Lading Description:

"Oxidizing Material - Ammonium Perchlorate - Keep Fire Away
 UFC #11 Item 22430
 NMFC #13 Item 43050"

c. Label Requirement:

Yellow Label (Per 49 CFR 173.13)
 For air shipment, apply Yellow Label as required by Rule No. 5 of NAVSUPPUB 505/MCO P4030.19

d. U.S. Coast Guard Class: Oxidizing Material (46 CFR 146.22-200 Table E)

e. Quantity Distance: Inert

f. Fire Fighting Specification (DD Form 836): Group I

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6. NOTES

6.1 Intended use. This material is intended for use as an oxidizer in solid propellants for the CCU-22/A Cartridge Impulse Assembly and the CKU-5/A Catapult Ejection Seat Assembly and other rocket motor applications.

6.2 Ordering data. Procurement documents should specify the following:

6.2.1 Procurement requirements.

a. Title, number, revision letter and date of this specification.

b. Quantity required.

* c. Whether tricalcium phosphate conditioner is required.

d. Place of inspection.

* e. Level of packaging and marking required (see 5.2 and 5.3).

f. Place of delivery

g. Amount of desiccant required (see 5.1.1).

* h. Safety precautions (see 6.3).

* 6.2.2 Contract data requirements. When this specification is used in a procurement which incorporates a DD Form 1423 and invokes the provisions of 7-104.0(n) of the Armed Services Procurement Regulations, the data requirements identified below will be developed as specified by an approved Data Item Description (DD Form 1664) and delivered in accordance with the approved Contract Data Requirements List (DD Form 1423) incorporated into the contract. When the provisions of ASPR-7-104.9(n) are not invoked, the data specified below will be delivered by the contractor in accordance with the contract requirements. Deliverable data required by this specification is cited in the following paragraph:

PARAGRAPH	DATA REQUIREMENTS	APPLICABLE DID
4.4	Test report	DI-T-3721

(Copies of data item descriptions required by the contractor in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer).

6.3 Safety precautions. The safety precaution requirements of the "Contractors' Safety Manual for Ammunition, Explosives, and Related Dangerous Material" (DOD 4145.26M) are applicable and should be specified in the contract or order as required by the Armed Services Procurement Regulations (ASPR) 1-323.

NOTE: When this specification is used as part of the description of work to be accomplished by a Government activity, the safety precaution requirements of "Ammunition and Explosives Ashore" (OP 5) should be made applicable.

- * 6.3.1 Explosive hazard classification. The Bureau of Explosives has determined that when 50% by weight or more of ammonium perchlorate is 15 μm or smaller particles, the ammonium perchlorate is a high explosive and must be handled, packed and marked accordingly. The contracting officer is directed to section 5 herein and NAVSEAINST 8023.1 for the explosive hazard classification. Certain classes of MIL-A-82667/1 and MIL-A-82667/2 meet this criterion.
- * 6.3.2 Hazard notice. The ammonium perchlorate described herein is an oxidizing material and/or high explosive and consequently presents a hazard in manufacture, handling, storage and shipment. The contractor should recognize this hazard and take appropriate measures to guard and protect against fire, explosion, adverse environment, corrosive atmosphere, rough handling and electrically induced incidents.
- * 6.4 Material safety data. Material safety data sheet requirements are applicable and should be specified in the contract as required by the Armed Services Procurement Regulations (ASPR) 1-323.2.
- * 6.5 Marginal identification The margins of this specification are marked with an asterisk to indicate where changes (additions, modifications, corrections, deletions) from the previous issue were made. This was done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in these notations. Bidders and suppliers are cautioned to evaluate the requirements of this document based on the entire content irrespective of the marginal notations and relationship to the last previous issue.

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