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2 September 1965

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MILITARY SPECIFICATION

AMMONIUM PERCHLORATE, TECHNICAL

This specification has been approved by the Department of Defense and is mandatory for use by the Departments of the Army, the Navy, and the Air Force.

1. SCOPE

1.1 Scope. This specification covers three grades and seven classes of ammonium perchlorate, (NH_4ClO_4).

1.2 Classification. The material shall be of the following grades and classes as specified in the contract or order (see 3.2 and 3.3).

Grade A - Low moisture content.
Grade B - Extra low moisture content.
Grade C - Tricalcium phosphate conditioner added.
Class 1 - Through 420 and 297 micron sieve, retained on 74 micron sieve.
Class 2 - Through 297 micron sieve.
Class 3 - Through 149 micron sieve
Class 4 - Graduated sieving.
Class 5 - Through 297 micron sieve, retained on 105 micron sieve.
Class 6 - Graduated sieving.
Class 7 - Graduated sieving.

2. APPLICABLE DOCUMENTS

2.1 Government 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:

/ FSC: 6810 /

SPECIFICATIONS

Federal

L-P-378	Plastic Film (Polyethylene, Thin Gage)
L-T-100	Tape -Pressure Sensitive Adhesive, Polyester Film
TT-E-485	Enamel, Semi-Gloss, Rust Inhibiting
PPP-D-729	Drums, Metal, 55 gallons (for shipment of Noncorrosive Materials)

Military

MIL-P-116	Preservation, Methods of
MIL-D-3464	Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification

STANDARDS

Federal

FED-STD-595	Colors

Military

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-109	Quality Assurance Terms and Definitions
MIL-STD-129	Marking for Shipment and Storage
MIL-STD-286	Propellants, Solid: Sampling, Examination and Testing
MIL-STD-1234	Pyrotechnics: Sampling, Inspection and Testing
MIL-STD-1235	Single and Multilevel Continuous Sampling Procedures and Tables for Inspection by Attributes

(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 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

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

(The Interstate Commerce Commission Regulations are now a part of the Code of Federal Regulations (1949 Edition and Revisions) available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Orders for the above publication should cite "49 CFR 71-90 (latest revision)").

3. REQUIREMENTS

3.1 Workmanship and Quality. Ammonium perchlorate shall be an odorless, white solid, free of visible impurities.

3.2 Chemical properties. Ammonium perchlorate shall conform to the chemical requirements shown in Table I when tested as specified in 4.3.

3.3 Granulation. Ammonium perchlorate shall conform to the granulation requirements shown in Table II when tested as specified in 4.3.10.

TABLE I - CHEMICAL REQUIREMENTS

Requirement	Percent by Weight			Test Method
	Grade A	Grade B	Grade C	
Total Moisture, maximum (Max)	0.08*	0.05	0.08*	4.3.1.2
Surface Moisture, max	0.020	0.015	0.020	4.3.1.3
Water Insoluble, max	0.03	0.01	0.25	4.3.2
Ash, Sulfated, max	0.25	0.15	0.45	4.3.3
Chlorate, as NH_4ClO_3, max	0.02	0.02	0.02	4.3.4
Chloride, as NH_4Cl, max	0.15	0.10	0.15	4.3.5
Assay, NH_4ClO_4, minimum (min)	99.0	99.0	98.8	4.3.6
Sodium and Potassium, max	0.08	0.05	0.08	4.3.7
Tricalcium Phosphate, as $Ca_3(PO_4)_2$	---	---	0.15-0.22	4.3.11
Iron as Fe_2O_3, max (When specified)	0.0036	0.0036	0.0036	4.3.8
pH (range)	(4.3-5.3)	(4.3-5.3)	(5.5-6.5)	4.3.9

*Class 7 may be 0.13% max.

TABLE II - GRANULATION

U.S. Sieve Number	Opening, Microns	Fraction Passing Through Sieve, %						
		Class 1	2	3	4	5	6	7
12	1680	-	-	-	-	-	-	99.5 min
18	1000	-	-	-	99.5 min	-	-	-
30	595	-	-	-	-	-		95 min
40	420	99.5 min	-	-	-	-		45 to 65
50	297	85 to 98	93 min	-	88 to 96	99.0 min	89 to 97	0 to 3
70	210	-	-	-	50 to 70	-	-	
100	149	-	-	70 min	17 to 38		18 to 50	-
140	105	-	-	-	2 to 15	1.0 min	2 to 15	-
200	74	1-9	-	-	1 to 5	-	-	-
325	44	-	-	-	2 max	-	-	-

4. QUALITY ASSURANCE PROVISIONS

4.1 General quality assurance provisions. The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. 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 Standard MIL-STD-109 in order to define the terms used herein. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.

4.1.1 Contractor quality assurance system. If the contractor desires to utilize a quality assurance system, which is at variance with the quality assurance provisions of 4.2 and 4.3 and other documents referenced herein, he shall submit a written description of the system to the contracting officer for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections to be performed, inspection stations, sampling procedures, methods of inspection (measuring and testing equipment), and provisions for control and disposition of non-conforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance provisions required by the provisions of 4.2 and 4.3 and the other documents referenced herein. The contractor shall not be restricted to the inspection station nor the method of inspection listed in this specification provided that an equivalent control is included in the approved quality control procedure. In cases of dispute as to whether certain procedures of the contractor's system provide equal assurance, the comparable procedure of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any changes to the written procedure that affects the degree of assurance required by this specification or other documents referenced herein.

4.1.2 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 submitted:

(a) A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.

(b) Quantity of product inspected.

(c) Results obtained for all inspection performed.

(d) Specification number and date together with an identification and date of changes.

(e) Certificates of analysis on all material procured directly by the contractor when such material is controlled by Government specifications listed in any of the contractual documents.

(f) Quantity of product in the lot.

(g) Date submitted.

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.

4.1.3 Government verification. Using the contractor's written quality assurance procedure (see 4.1.1), this specification, and other contractual documents as a guide, the Government inspector shall verify all quality assurance operations performed by the contractor. Verification shall be in accordance with a or b as applicable, the decision being the responsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all quality assurance provisions of other specifications referenced in any of the contratual documents have been complied with. Deviations from prescribed or agreed upon procedures discovered by the Government inspector shall be brought to the attention of the supplier. Disposition of the product and remedial action shall be as directed by the Government inspector and, depending on the nature of the deviation, may consist of lot rejection, screening, re-sampling, re-instruction of the supplier's employees, or other appropriate action:

(a) Verification at the point of manufacture shall be accomplished at unscheduled intervals in accordance with 4.1.3.1 and 4.1.3.2.

(b) Verification at the point of delivery shall be in accordance with 4.1.3.2.

4.1.3.1 Surveillance. Surveillance shall include, but not be limited to:

(a) Observation of procedures concerning lot formation and identification.

(b) Observation of sampling procedures and application of acceptance criteria.

(c) Determination that all required examinations and tests are performed in accordance with the prescribed procedures of this specification, or approved equivalents thereto.

(d) Review of procedures for control and disposition of non-conforming material.

4.1.3.2 Product inspection. Product inspection shall consist of Government inspection of product which has been previously inspected by the contractor and found to meet the quality assurance provisions of this specification and that the contractors record are reliable.

4.2 Inspection provisions.

4.2.1 Lot formation. A lot shall consist of one or more batches of ammonium perchlorate, 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 perchlorate that has been subjected to the same unit chemical or physical process intended to make the final product homogeneous. The product shall be submitted for inspection in accordance with Standard MIL-STD-105 (or Standard MIL-STD-1235 when applicable).

4.2.2. Examination. Sampling plans and procedures for the following classification of defects shall be in accordance with MIL-STD-105. Continuous sampling plans in accordance with Standard 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 for each major defect and AQL of 0.65 for each minor defect

4.2.2.1 Containers for domestic shipment and limited storage, prior to closing.

Categories	Defects	Method of Inspection	Code No. (See 6.7)
Critical:	None defined		
Major:	AQL 1.00 percent	Major	
101.	Thickness of liners incorrect minimum (min)	Gage	01001
102.	Liner material improper	Visual	01002
103.	Liner missing, cut, torn or punctured	Visual	01003
104.	Liner improperly sealed	Visual	01004
105.	Desiccant bags missing	Visual	01005

4.2.2.2 Container for domestic shipment and limited storage, closed.

Categories	Defects	Method of Inspection	Code No. (see 6.7)
Critical:	None defined		
Major:	AQL 0.65 percent	Major	
101.	Container poorly sealed	Visual	02001
102.	Marking misleading or unidentifiable	Visual	02002
103.	Weight incorrect	Scale	02003
Minor:	None defined		

4.2.2.3 Container for overseas shipment, prior to closing.

Categories	Defects	Method of Inspection	Code No. (see 6.7)
Critical:	None defined		
Major:	AQL 1.00 percent	Major	
101.	Thickness of liner, min.	Gage	03001
102.	Liner material improper	Visual	03002
103.	Liner missing, cut, torn or punctured	Visual	03003
104.	Liner improperly sealed	Visual	03004
105.	Desiccant bags missing	Visual	03005
Minor:	None defined		

4.2.2.4 Container for overseas shipment, closed:

Categories	Defects	Method of Inspection	Code No. (see 6.7)
Critical:	None defined		
Major:	AQL 0.25 percent	Major	
101.	Weight incorrect	Scale	04001
102.	Marking misleading or unidentifiable	Visual	04002
Minor:	None defined		

4.2.3 Testing.

4.2.3.1 Sampling by lot. Samples shall be taken at random from each lot as follows:

Containers in Lot	Minimum No. of Containers Sampled
1-9	All
10-99	10
100 and over	10 percent

4.2.3.1.1 Primary sample. A sample thief, 3/4 inches in diameter and of sufficient length to reach the bottom of the container, shall be inserted at an oblique angle to the maximum possible depth in each container sampled. Insertion of the thief shall be repeated at different locations until a pint of sample is removed. Samples shall be placed in clean, dry, moisture proof containers with a screw or compression type seal. Each sample container shall be labeled with the following data:

(a) Material name
(b) Lot number
(c) Type and Granulation Class
(d) Container from which sample was taken
(e) Manufacturer
(f) Date

4.2.3.1.2 Composite sample. The primary samples shall be divided into two groups. A one pint composite sample shall be prepared for each group by thoroughly mixing equal portions of each primary sample and transferring this mixture to clean, dry, moisture proof containers. The containers shall then be labeled with the following data:

(a) Material name
(b) Lot number
(c) Type and Granulation Class
(d) Composite Sample
(e) Manufacturer
(f) Date

4.3 Test Methods and procedures. Distilled or deionized water and American Chemical Society reagent grade chemicals shall be used throughout the tests. Tests shall be conducted as follows:

4.3.1 Moisture - (Code No. 05001). The Karl Fischer (KF) method shall be used with any suitable apparatus according to the general procedure described below (see 6.5.5). The end point may be determined either visually or electrometrically by means of either direct or back titration. In all operations, protect the sample against change in moisture content.

4.3.1.1 Standardization of Karl Fischer Reagent. Add 100 milliliters (ml) of the pyridine-methanol solution (see 6.5.3) to a clean, dry titration flask containing a stirring bar. Close the flask, start the magnetic stirrer and titrate to an end point, using either a direct or back titration. Add 0.10 ± 0.01 grams (gm) of sodium tartrate dihydrate (see 6.5.4), keeping the flask open for the minimum time possible, and allow to stir until solution is practically complete. Add small increments of Karl Fischer Reagent (KFR) to assist solution and titrate to an end point after solution is complete. For back titration, a standard solution of water in-methanol (WM) should be used (see 6.5.2). Calculate the water equivalence (F) of the KFR as follows:

	Direct Titration		Back Titration
$F =$	$\frac{0.1556W}{A}$	$=$	$\frac{0.1566W}{AR-B}$

Where:

A = Volume of KFR required in direct titration or added for back titration, ml.

B = Volume of WM solution used in back titration, ml.

R = Ratio of volume of standard WM used in back titration (A) to volume of KFR added (B). $\left(R=\frac{A}{B}\right)$

W = Weight of sodium tartrate dihydrate, gm.

The ratio, R, is determined by adding an accurately measured volume of approximately 10 ml of KFR to a convenient volume of the methanol-pyridine solvent (see 6.5.3) which has just been brought to an end point. Titrate the KFR with standard WM solution (see 6.5.2).

4.3.1.2 Total moisture. - (Code No. 06001) Place approximately 15 gm portions of the sample in dry, stoppered weighing bottles and determine the total weight of sample and bottle to the nearest 10 milligrams (mg). Add 100 ml of methanol-pyridine solution to the titration vessel and titrate to an end-point with KFR. As soon as the end-point is reached, transfer the contents of the weighing bottle to the titration vessel and immediately reclose the vessel. Do not take time to try to transfer all of the sample, but restopper the weighing bottle and again weigh to the nearest 10 mg. Stir the contents of the titration vessel until the sample is dissolved. If necessary, small amounts of KFR may be added to facilitate solution. For direct titration, titrate to an end-point with KFR. For back titration, add an excess of KFR and back titrate with WM. Calculate the percent water as follows:

	Direct Titration		Back Titration
Percent Water =	$\frac{FA100}{W}$	=	$\frac{F\,(AR-B)\,100}{W}$

Where:

A = KFR required for direct titration, or used in back titration, ml.

B = WM solution used in back titration, ml.

F = Water equivalent of KFR, gm/ml.

W = Weight of specimen, gm.

4.3.1.3 Surface or external moisture. - (Code No. 07001) Proceed as specified in 4.3.1.2 except that benzene shall be used in place of the methanol pyridine mixture and titrating as soon as the sample has been transferred to the flask.

4.3.2 Water insoluble. - (Code No. 08001) Transfer a specimen of about 50 gm (weighed to the nearest 0.1 gm) to a 400 ml beaker and add about 250 ml of water. Heat to $75^{\circ}\pm5$ degrees Centigrade ($^{\circ}$C) and stir until the salt is completely dissolved. Filter the contents of the beaker through a tared, fine porosity, sintered glass crucible and transfer any residue from the beaker to the crucible

by washing thoroughly with additional water heated to the same temperature as above. Dry the crucible and contents for 2 hours at $100^{\circ} \pm 5^{\circ}C$, cool in a desiccator and weigh. Calculate the water insoluble content of the sample as follows:

$$\text{Percent water insoluble} = \frac{100\,W'}{W}$$

Where:

W' = Weight of residue, gm

W = Weight of sample, gm

4.3.3 Sulfated Ash. - (Code No. 09001) Ignite a clean porcelain, silica or platinum crucible and cover for approximately 30 minutes at $800^{\circ} \pm 25^{\circ}C$. Cool in a desiccator, weigh to the nearest 0.5 mg, and add approximately 10 gm of sample weighed to nearest 0.01 gm. Add 3 ml of concentrated sulfuric acid, place the cover on upside down and heat over a bunsen burner or hot plate until most of the sample is burned off. (Caution: Heat the sample in a hood and keep a safety shield interposed between the operator and the sample. Heat gently since NH_4ClO_4 may decompose violently if heated over $300^{\circ}C$). When volatilization of the sample is essentially complete, transfer the crucible with inverted cover to a muffle furnace, and ignite at $800^{\circ} \pm 5^{\circ}C$ for at least 30 minutes, and transfer to a desiccator. Replace the crucible cover in an upright position, cool and weigh to the nearest 0.5 mg. Calculate the sulfated ash content as follows:

$$\text{Percent sulfated ash} = \frac{100\,W'}{W}$$

Where:

W' = Weight of residue, gm

W = Weight of sample, gm

4.3.4 Chlorate - (Code No. 10001).

4.3.4.1 Orthotolidine reagent. Dissolve 1.35 gm of o-tolidine dihydrochloride in 500 ml of water. Add this solution, with constant stirring, to a mixture of 350 ml of water and 150 ml of concentrated hydrochloric acid. This reagent should be stored at room temperature in amber bottles or in the dark, protected from sunlight and contact with rubber. The reagent must be discarded after 6 months.

4.3.4.2 Standard solution. Dissolve 0.212 gm of sodium chlorate in water and dilute to one liter.

4.3.4.3 Procedure. Transfer 2.00 gm of the specimen and 1.8, 1.9, 2.0 and 2.1 ml aliquotes of the standard solution to separate, glass stoppered Erlenmyer flasks. Add 50 ml of water and 20 ml of hydrochloric acid. Maintain at 40°C for 30 minutes, cool to room temperature, and transfer to 100 ml Nessler tubes containing 5 ml of o-tolidine reagent. Dilute to 100 ml with water, mix and allow the color to develop for 5 minutes, preferably in the dark. Compare the color with the standards and calculate the ammonium chlorate content on the basis of the matching standard as follows:

Percent $NH_4ClO_3 = 0.01A$

Where:

A = Volume of aliquot in matching standard, ml.

Note: Permanent standards may be prepared from copper sulfate and potassium dichromate (see 6.4).

4.3.5 Chloride. - (Code No. 11001) Dissolve 20 gm of the specimen (weighed to the nearest 0.05 gm) in 150 ml of water in an Erlenmeyer flask and add 3 to 4 ml of nitric acid. Add standardized 0.05 normal (N) silver nitrate solution (see 6.6.1) from a buret until the precipitate coagulates, mix thoroughly, allow to stand a moment and add a little more silver nitrate solution to the clear supernatant liquid. If precipitation is complete (no marked cloudiness occurs) add an additional 5 ml of the silver nitrate solution. If precipitation is incomplete, add more silver nitrate. Mix, add 1 to 2 ml of nitrobenzene and mix again. The supernatant liquid must not be milky. Add 2 to 3 ml of ferric alum indicator (see 6.6.3) solution and titrate with 0.05 N potassium thiocyanate solution (see 6.6.2) to a permanent red-brown color.

Alternatively, chloride may be determined by direct potentiometric titration. If this method is selected, prepare the specimen as in the preceding paragraph, and titrate with 0.05 N silver nitrate solution to a permanent end point using a silver indicator electrode and a glass reference electrode.

Calculate the ammonium chloride content as follows:

$$\text{Percent ammonium chloride} = \frac{5.35\,(aA - bB)}{W} \text{ (Back Titration)} = \frac{5.35\,aA}{W} \text{ (Direct Titration)}$$

Where:

A = Volume of silver nitrate solution, ml.

B = Volume of potassium thiocyanate solution, ml.

a = Normality of silver nitrate solution.

b = Normality of potassium thiocyanate solution.

W = Weight of sample, gm.

4.3.6 Assay. - (Code No. 12001) Transfer an accurately weighed portion of approximately 0.5 gm of the specimen to a platinum crucible containing a known weight of approximately 5 gm of sodium carbonate and mix carefully. Overlay the mix with a known weight of sodium carbonate (approximately 1 gm) so that a correction can be made for any chloride in the sodium carbonate. Fuse the mixture over a low flame for approximately 30 minutes and then gradually increase the flame to full heat. Dissolve the fusion in a covered beaker containing 50 ml of water and add 50 ml of 1 nitric acid : 1 water. Quantitatively transfer this solution to a 500 ml Erlenmeyer flask and dilute to about 150 ml with water. Add exactly 50 ml of 0.1 N silver nitrate solution (see 6.6.1), mix well, add 2 ml of nitrobenzene and mix again. The supernatant liquid must not be milky. Add 2 to 3 ml of ferric alum indicator solution (see 6.6.3) and back titrate with 0.1 N potassium thiocyanate solution (see 6.6.2) to the first detectable color change which persists for at least 30 seconds.

Alternatively, perchlorate may be determined by a direct electrometric titration. If this method is selected, fuse the sample with sodium carbonate and dissolve in nitric acid as directed above. Then titrate with 0.1 N silver nitrate solution to a permanent end-point using a silver indicator electrode and a glass reference electrode.

With either method, conduct a blank determination to correct for titratable impurities. Calculate the ammonium perchlorate content as follows:

Back Titration

$$\text{Percent ammonium perchlorate} = \frac{11.75\ N\ (A-B)}{W} - (2.20\ C + 1.16\ D)$$

Direct Titration

$$= \frac{11.75\ N'\ (A'-B')}{W} - (2.20\ C + 1.16\ D)$$

Where:

N = Normality of potassium thiocyanate.

N' = Normality of silver nitrate.

A = Volume of potassium thiocyanate required for blank titration, ml.

A' = Volume of silver nitrate required for sample titration, ml.

B = Volume of potassium thiocyanate required for sample titration, ml.

B' = Volume of silver nitrate required for blank titration, ml.

C = Percent ammonium chloride (see 4.3.5)

D = Percent ammonium chlorate (see 4.3.4)

W = Weight of specimen, gm.

4.3.7 Sodium (Na) and potassium (K). (Code No. 13001).

4.3.7.1 Preparation of reagents. Quantitatively transfer 1.9066 gm of potassium chloride and 2.5416 gm of sodium chloride into separate 1 liter volumetric flasks containing approximately 250 ml of water. Swirl to dissolve the salt and dilute to volume with water. Each solution contains 1 mg/ml of cation. From these stock solutions transfer aliquots to other volumetric flasks and dilute with water so that at least four standard solutions are prepared in the range 0.005 to 0.03 mg/ml for K ion and 0.001 to 0.005 mg/ml for Na-ion.

4.3.7.2 Preparation of standard curves. Determine the net emission in terms of absorbance or percent transmittance of the standard solutions using a wavelength of 766.5 milli-microns (mμ) for K-ion and 589 mμ for Na-ion. The minimum practical slit width should be used. Prepare a graph by plotting absorbance vs. concentration on linear coordinates or percent transmittance vs. concentration on semi-log coordinates. Fit a straight line to the points.

4.3.7.3 Analysis of Sample: Quantitatively transfer 4.0 gm of ammonium perchlorate, weighed to the nearest mg, into a 100 ml volumetric flask. Dissolve in distilled water and dilute to volume. Determine the absorbance or percent transmittance of each standard solution, a distilled water blank solution, and the sample solution, by flame photometry using the manufacturer's manual of instructions for the spectrophotometer. Read the concentration of the sample directly from the standard curve.

Calculate the potassium and sodium content as follows:

$$\text{Potassium and sodium, percent} = \frac{10\,(R_K + R_{Na})}{W}$$

Where:

R_K = Potassium concentration as read from standard curve, mg/ml.

R_{Na} = Sodium concentration as read from standard curve, mg/ml.

W = Weight of sample, gm/100 ml.

4.3.8 Iron - (Code No. 14001)

4.3.8.1 Preparation of standard iron solution (1 ml = 0.0001 gm Fe). Dissolve 0.8635 gm of ferric alum, $FeNH_4(SO_4)_2.12\,H_2O$ in 100 ml of sulfuric acid (H_2SO_4) solution prepared by cautious addition of 5 ml of H_2SO_4 to 95 ml of water. When solution is complete and at room temperature (20-25°C), dilute to 1 liter in a volumetric flask.

4.3.8.2 Procedure. Dissolve 7.00 gm of the specimen in 75 ml of water. Add 2.0 ml of concentrated H_2SO_4 and transfer to a 100 ml Nessler tube. Add 2 drops of 1 percent potassium permanganate solution and 10 ml of 2 percent ammonium thiocyanate. Dilute to 100 ml and mix well. Into a second Nessler tube containing the same amounts of water and reagents as above, pipette 1.75 ml of the standard iron solution. Dilute and mix well. If the unknown is no darker than the standard, the specimen meets the requirement for iron.

4.3.9 pH. - (Code No. 15001) Add 50 ml of freshly boiled water to 20.0 gm of specimen. Stir and cool to room temperature (20° to 25°C). Measure the pH on a suitable potentiometer having glass, calomel electrodes.

4.3.10 Granulation. - (Code No. 16001) The granulation shall be determined in accordance with Standard MIL-STD-1234, Method 201.1.

4.3.11 Tricalcium phosphate (TCP). - (Code No. 17001)

4.3.11.1 Preparation of reagents. (a) Ammonium Vanadate Solution - Add 2.350 gm of ammonium metavanadate to approximately 400 ml of warm distilled water in a 1 liter volumetric flask. Slowly and cautiously add 14 ml of 70-72 percent perchloric acid, mix to effect solution, cool to room temperature, and dilute to volume with water; (b) Ammonium Molybdate Solution - Dissolve 100 gm of molybdic acid in a mixture of 300 ml of distilled water and 80 ml of ammonium hydroxide. Filter the solution and boil the filtrate for 20 minutes. Cool, transfer to a 1 liter volumetric flask and dilute to volume with distilled water; (c) Perchloric Acid - 70-72 percent (American Chemical Society grade).

4.3.11.2 Preparation of calibration curve. Quantitatively transfer 5.000 gm of TCP to a 1 liter volumetric flask, add 500 ml of water and 20 ml of perchloric acid. Mix to dissolve and dilute to volume with distilled water. Transfer 1, 2, 3, 4 and 5 ml aliquots to separate 100 ml volumetric flasks. Add to each flask 25 ml of water and 17 ml of perchloric acid. Cool to room temperature and add 10 ml of ammonium vanadate solution. Dilute to 75 ml with water, mix and cool to room temperature, Add 10 ml of ammonium molybdate solution, dilute to volume with water and let stand at room temperature for at least 30 minutes. Prepare a blank solution containing all the reagents in the specified amounts but without the TCP. Using the blank solution in the reference cell, determine the absorbance of each of the five standard solutions at 470 mμ. Prepare a calibration curve by plotting absorbance vs. concentration, expressed as gm TCP/100 ml of solution, and draw the calibration curve by fitting the best straight line to the points.

4.3.11.3 Procedure. Quantitatively transfer 2.50 gm of sample to a 100 ml volumetric flask and dissolve in about 50 ml of water. Add, slowly and cautiously, 20 ml of perchloric acid and, after thorough mixing, cool to room temperature. Add 10 ml of ammonium vanadate solution, dilute to 75 ml with water, mix and cool to room temperature. Add 10 ml of ammonium molybdate solution, dilute to volume with water, mix well and allow to stand at room temperature for at least 30 minutes. Determine the absorbance of the sample solution at 470 mμ, using the blank solution in the reference cell. Determine the weight of the TCP by referring to the calibration curve and calculate the amount in the sample by substituting in the following equation:

$$\text{TCP, percent} = \frac{100\ A}{W}$$

Where:

A = Weight of TCP in sample, gm.
W = Weight of sample, gm.

4.4 Acceptance and rejection criteria. If any specimen does not meet the requirements of this specification, the lot represented shall be rejected.

5. PREPARATION FOR DELIVERY

5.1 Packing. Packing shall be level A or C (see 6.2).

5.1.1 Level A. Ammonium perchlorate shall be packed in 250 pound quantities in 30 gallon capacity drums conforming to 49 CFR 71-90, ICC specification 6A. The drums shall be cleaned, phosphate coated, and painted in conformance with the requirements of PPP-D-729. Paint shall conform to TT-E-485, color No. 24064 of Federal Standard 595. The drums shall be provided with bag liners fabricated from 2 mil (min) polyethylene conforming to L-P-378. The bag liners shall have flat or square bottoms with heat sealed seams that meet the requirements of MIL-P-116. The bag shall be closed either by pressure sensitive tape conforming to L-T-100 or by heat seal (Caution: Prevent contact of the ammonium perchlorate with the heat sealer). Two 8-unit bags of silica gel, conforming to MIL-D-3464, shall be inserted in each drum.

5.1.2 Level C. The ammonium perchlorate shall be packed in uniform quantities in conformance with the manufacturer's commercial practice, or as specified in the contract, to provide protection against damage during shipment from the supply source to the first receiving activity for immediate use. Containers shall comply with ICC Regulations, Uniform Freight Classification Rules or other carrier regulations applicable to the mode of transportation. Containers for like class material shall be of uniform size, shape, and material. Containers shall be provided with a polyethylene liner as specified in 5.1.1.

5.2 MARKING

5.2.1 Level A. In addition to any special marking required by the contract or order, all shipping containers packed in accordance with level A shall be marked in accordance with MIL-STD-129 and ICC regulations.

5.2.2 Level C. In addition to any special marking required by the contract or order, all shipping containers shall be marked in accordance with the manufacturer's commercial practice and ICC regulations.

6. NOTES

6.1 Intended Use.

6.1.1 Grade A. This ammonium perchlorate is intended for use in rocket propellants, in tracer ammunition, and in flame throwers.

6.1.2 Grades B and C. This ammonium perchlorate is intended for use in rocket propellants.

6.2 Ordering data. Procurement documents should specify:

a. Title, number and date of this specification.
b. Grade and class required. (see 1.2 and 3.3).
c. Level of packing required.

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

6.4 Chlorate test. For further information on the o-tolidine reagent and permanent standards for chlorate test (see 4.6.4), refer to the Standard Methods for the Examination of Water and Waste Water, 11th ed., p. 85-91, published by the American Public Health Association, 1790 B'way, New York 10, N.Y..

6.5 Total moisture determination. The solutions and standards required for the Karl Fischer tests may be prepared as follows:

6.5.1 Karl Fischer Reagent. Dilute stabilized KFR (water equivalence 5-7 mg/ml) with absolute methyl alcohol (0.01 percent water max.) or stabilized KF diluent, so that the water equivalence is 1.4-2.3 mg/ml.

6.5.2 Water-in-Methanol solution. Add sufficient water to absolute methanol so that the final concentration is approximately 1 mg/ml.

6.5.3 Pyridine-Methanol solution. Mix 3 volumes of anhydrous pyridine with 1 volume of anhydrous methanol. If necessary, the solvents can be made anhydrous (0.01% H_2O, max) by the methods given below.

6.5.3.1 Pyridine. Add 1 volume of benzene to 19 volumes of pyridine and distill until 5 percent of distillate has been collected. Discard the distillate and use the dry residual 95 percent.

6.5.3.2 Methanol. Distill the methanol over magnesium.

6.5.4 Sodium Tartrate Dihydrate standard. The water content of the standard may be verified by drying a 2 to 3 gm sample in a wide-mouth weighing bottle at 155 $\pm$ 5°C for 4 hours. The loss in weight should not be less than 15.61 percent nor more than 15.71 percent.

6.6 Standard and indicator solutions. The solutions required for the determination of chloride content (see 4.3.5) and assay (see 4.3.6) may be prepared as follows:

6.6.1 Silver Nitrate solutions. The 0.1N solution (nominal) may be prepared as described in MIL-STD-1234, method 607.1. An aliquot of this solution may be diluted with an equal volume of water to give a nominal 0.05N solution.

6.6.2 Potassium Thiocyanate solutions. The 0.1N solution (nominal) may be prepared and standardized as described in MIL-STD-1234, method 608.1. An aliquot of this solution may be diluted with an equal volume of water to give a nominal 0.05N solution.

6.6.3 Ferric alum indicator. This solution may be prepared from Fe NH_4 (SO_4). 12 H_2O as described in MIL-STD-286 method 705-1.

6.7 Inspection code numbers. The five digit code numbers assigned to the inspections herein are to facilitate future data collection and analysis by the Government.

CUSTODIANS:

Army - MU

Navy - Wp

Air Force - 12

PREPARING ACTIVITY:

Army - MU

Project No. 6810-0241

To detach this form, cut along this line

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.

SPECIFICATION

ORGANIZATION | 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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1 APR 63

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