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30 September 1964
SUPERSEDES
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MILITARY SPECIFICATION

SODIUM CARBOXYMETHYL CELLULOSE (NaCMC) (FOR AMMUNITION USE)

1. SCOPE

1.1 This specification covers the minimum requirements for sodium carboxymethyl cellulose (NaCMC) as the granulating agent in the manufacture of lead azide.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

FEDERAL

PPP-D-723 - Drums, Fiber
PPP-D-729 - Drums, Metal, 55 Gallon (for shipment of noncorrosive material)

MILITARY

MIL-P-112 - Packaging and Packing for Overseas Shipment Drums, Plywood (for drums whose weight of contents does not exceed 200 pounds)
MIL-I-45208 - Inspection Requirements; General Specification for.

STANDARDS

MILITARY

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

FSC 6810

(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 shall apply.

American Society for Testing and Materials (ASTM) -
ASTM Standards
ASTM-D-1439-64T, "Tentative Methods of Testing Sodium Carboxymethylcellulose"
(Copies may be purchased directly from ASTM, 1916 Race Street, Philadelphia 3, Pa.)

UNIFORM FREIGHT CLASSIFICATION PUBLICATION
(Application for copies of these freight classification rules should be addressed to the Consolidated Classification Committee, 202 Chicago Union Station, Chicago 6, Ill.)

AMERICAN TRUCKING ASSOCIATION PUBLICATION
National Motor Freight Classification Rules and Container Specifications.
(Application for copies should be addressed to the American Trucking Association, 1424 16th Street N.W., Washington, D.C.)

3. REQUIREMENTS

3.1 Material.-The material used in the manufacture of sodium carboxymethyl cellulose shall be such as to produce a finished product conforming to the requirements stated in Table I when determined as specified in the applicable paragraphs of section 4.3.

Table I

Color and form.-White to cream colored powder free from gritty particles, visible impurities and foreign matter when tested as specified in 4.3.1.
Moisture.-10.0 percent maximum (max.) when tested as specified in 4.3.2.
Viscosity.-A 2 percent solution at 25 degrees C. - 300 centipoises (cps.) minimum (min.) and 750 cps. max. when tested as specified in 4.3.3.
Sodium chloride.-4.0 percent max. when tested as specified in 4.3.4.
Sulfated Ash.-20 percent max. when tested as specified in 4.3.5.

Insoluble Matter.-10 percent max. when tested as specified in 4.3.6.
pH Value.-6 min. and 8 max. when tested as specified in 4.3.7.
Degree of Etherification (D.E.).-0.45 min. and 0.63 max. when tested as specified in 4.3.8.
Purity.-90.0 percent min. when tested as specified in 4.3.9.

3.2 Workmanship.-Acids shall not be used to alter the pH value of the sodium carboxymethyl cellulose (NaCMC).

4. QUALITY ASSURANCE PROVISIONS

4.1 General quality assurance provisions.-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. Reference shall be made to Standard MIL-STD-109 in order to define terms used herein. The provisions of Specification MIL-A-2550 shall apply.

4.1.1 Contractor inspection system.-The contractor shall provide and maintain an effective inspection system in compliance with the requirements of specification MIL-I-45208. If due to the nature of the manufacturing process, the contractor desires to utilize a quality assurance system which is at variance with the quality assurance provisions of Specification MIL-I-45208 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 or the method of inspection listed in this specification provided that an equivalent control is included in the approved quality assurance 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 effects 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. Number of units 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. Number of batches 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, this detail specification, the applicable drawings and other contractual documents as a guide, the Government inspector shall verify at unscheduled intervals all quality assurance operations performed by the contractor. Verification will be in accordance with Specification MIL-I-45208 and will be performed to the extent necessary to assure compliance with the contractual requirements. Severity of Government inspection of individual characteristics will be directly related to the seriousness of the classification assigned. In no instance will a characteristic classified "critical" be accepted solely on the basis of contractor's records.

4.2 Inspection provisions

4.2.1 Lot formation.-A lot shall consist of one or more batches of sodium carboxymethyl cellulose, 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 sodium carboxymethyl cellulose 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 Standard 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 0.25 percent for each major defect and an AQL of 0.40 percent for each minor defect.

4.2.2.1 Drums, prior to filling (Polyethylene liner).

Categories	Defects	Method of Inspection	Code No. (see 6.3)
Critical: None defined.			
Major:	AQL 0.25 percent		
101.	Liner cut, torn or punctured..	Visual	01001
Minor: None defined.			

4.2.2.1.1 Drums, prior to filling (alternative method).

Critical: None defined.			
Major:	AQL 0.25 percent		
101.	Laminations or coatings incomplete.....	Visual	01002
Minor: None defined.			

4.2.2.2 Drums, prior to closing (Polyethylene liner)

Categories	Defects	Method of Inspection	Code No.
Critical: None defined.			
Major:	AQL 0.25 percent		
101.	Liner not completely sealed...	Visual	02001
Minor: None defined.			

4.2.2.3 Drum, sealed (steel)

Categories	Defects	Method of Inspection	Code No.
Critical: None defined			
Major:			
101.	Closing ring damaged or not properly positioned so it does not close drum properly.....	Visual	03001
Minor:	AQL 0.40 percent		
201.	Marking misleading or unidentifiable.....	Visual	03002

4.2.2.4 Drum, sealed (fiber)

Categories	Defects	Method of Inspection	Code No.
Critical: None defined			
Major:	AQL 0.25 percent		
101.	Weight of contents.....	Scale	04001
102.	Closure incomplete or damaged to the extent that contents sift out.....	Visual	04002
Minor:	AQL 0.40 percent		
201.	Markings misleading or unidentifiable.....	Visual	04003

4.2.2.5 Drum, sealed (plywood)

Categories	Defects	Method of Inspection	Code No.
Critical: None defined.			
Major:	AQL 0.25 percent		
101.	Weight of contents.....	Scale	05001
102.	Strapping missing or broken...	Visual	05002

Minor:	AQL 1.0 percent		
201.	Strapping mislocated.....	Visual	05003
202.	Marking misleading or unidentifiable.....	Visual	05004

4.2.3 Determination of color and form, moisture, viscosity, sodium chloride, sulfated ash, insoluble matter, pH value, degree of Etherification and purity (see 3.1 and 4.3.1 to 4.3.9) Major defect.

4.2.3.1 Testing

4.2.3.2 Sampling by lot.-A random representative sample of approximately three ounces from each of 10 containers shall be selected from each lot. When lots are comprised of 10 containers or less, each container shall be sampled to form a 30 ounce composite sample.

4.2.3.2.1 Preparation of composite.-Approximately three ounce samples of sodium carboxymethyl cellulose shall be removed from each container of the ten containers in order to obtain a thirty ounce sample. If there are less than 10 containers, equal primary samples in sufficient quantity to total thirty ounces, shall be removed from each container. The individual primary samples shall then be combined in order to form a homogeneous composite sample of thirty ounces and subjected to the tests specified in 4.3. If the composite sample fails to comply with any of the requirements specified, the lot shall be rejected on a major defect basis.

4.3 Test methods and procedures

4.3.1 Color and physical form.-Code Number 06001.-Determine the color and physical form by visual examination.

4.3.2 Moisture content.-Code Number 07001

4.3.2.1 Determine the moisture content in accordance with the method described in ASTM-D-1439-64T.
(NOTE: Save this sample for the determination described in 4.3.5. Do not use this sample for any other determinations listed below).

4.3.3 Viscosity.-Code Number 08001

4.3.3.1 Determine the viscosity in accordance with the method described in ASTM-D-1439-64T.

4.3.4 Sodium chloride content.-Code Number 09001

4.3.4.1 Determine the sodium chloride content in accordance with the method described in ASTM-D-1439-64T.

4.3.5 Sulfated Ash.-Code Number 10001

4.3.5.1 Apparatus

Porcelain (Ignition) Crucible
Hotplate
Muffle Furnace
Desiccator

4.3.5.2 Reagents

Nitric Acid, 70 percent, A.C.S. grade
Sulfuric Acid, 95 percent, A.C.S. grade

4.3.5.3 Accurately weight a 3 to 5 gm. sample of the dried material obtained from 4.3.2 into a tared porcelain (ignition) crucible. Place crucible and contents into a furnace maintained at 600 degrees plus or minus 50 degrees C. for 15 minutes. Remove from furnace and cool. Add 2 ml. of concentrated sulfuric acid and 5 drops of 70 percent nitric acid to the crucible. Place crucible and contents on a hot plate until acid is completely evaporated. Remove crucible from hot plate and again place in furnace maintained at 600 degrees plus or minus 50 degrees C. Allow to remain in furnace for 1 hour. Remove from furnace, cool in a desiccator and weigh. Calculate the sulfated ash content as follows;

$$\text{Percent Sulfated Ash} = \frac{A \times 100}{B}$$

Where:
A = weight of ash
B = gms. of specimen

4.3.6 Insoluble matter.-Code Number 1101.

4.3.6.1 Apparatus

8 oz. screw-top jars to fit in centrifuge
Centrifuge - International Size 1, Type C or equivalent.

4.3.6.2 Procedure

Transfer a 4 gram sample of the NaCMC weighed to the nearest milligram to an 8 oz. bottle and add 200 ml. of distilled water. Mark the bottle with a crayon at the liquid level. Stir the mixture with a mechanical stirrer until the sample is completely dispersed (usually about 30 minutes). Put the screw cap on the bottle and centrifuge for 30 minutes at 1500 rpm. Siphon off as much as possible of the supernatant liquid without disturbing the sediment. Add water to the mark, cap the bottle, and shake vigoriously to disperse the insoluble matter. Centrifuge again for 30 minutes at 1500 rpm. Proceed as before until the sample has been centrifuged four times. After the final centrifugation and removal of supernatant liquid, transfer quantitatively the insoluble matter to a tared evaporating dish with the aid of a distilled water wash bottle. Evaporate most of the water on a steam bath and the residue in an oven to constant weight at 100 degrees C. plus or minus 2 degrees C. Cool and weigh.

Calculate the insoluble matter as follows:

$$\text{Percent water insoluble matter} = \frac{10{,}000\ A}{W\ (100 - M)}$$

Where:

A = weight of dry residue
W = weight of sample
M = percent moisture found in sample (4.3.2)

4.3.7 pH Value.-Code Number 12001.-In a 100 ml. beaker dissolve a 1 gm. specimen of NaCMC (dry basis) in 100 ml. of distilled water. Determine the pH at room temperature using an A. Beckman pH meter, or an approved equal.

4.3.8 D.E., - Code Number 13001

4.3.8.1 Apparatus

a. Mechanical pulverizer - Micro pulverizer, Type CF, manufactured by Pulverizing Machine Co, Summit, N.J. or Filzwill Model JT manufactured by W. J. Fitzpatrick Co, Chicago, Ill. have been found satisfactory.

b. Air drive stirring apparatus.-A.H. Thomas Catalogue Number 9224, with glass propeller has been found satisfactory.

4.3.8.2 Reagents

a. Diphenylamine reagent - dissolve 0.5 gm. diphenylamine in 120 ml. 85 percent sulfuric acid.

b. 0.5 Molar nitric acid - 26 ml. concentrated nitric acid made up to a liter with distilled water.

c. 95.0 percent ethyl alcohol.

4.3.8.3 Grinding.-Grind the specimen on a mechanical pulverizer so that it passes through a 10 mesh sieve.

4.3.8.4 Procedure.-To a 250 ml. beaker containing 100 ml. of 95 percent ethyl alcohol, add 5 gm. of the NaCMC and agitate with a mechanical stirrer. Add slowly to the slurry, while stirring, 45 ml. distilled water and continue to stir for at least 5 minutes. Allow the solids to settle and decant the supernatant liquid. Add 100 ml. of 95 percent ethyl alcohol and slurry for about 2 minutes. Allow the solids to settle and decant the supernatant liquid. Add 100 ml. of 95 percent ethyl alcohol and, while stirring, add 5 ml. concentrated nitric acid and continue agitation for 2 minutes. Heat and slurry on a steam bath and boil for 5 minutes, remove and agitate again for at least 10 minutes. When the stirring is complete, filter through a Buchner funnel (No. 1 Whatman filter paper). Decant all the liquid through the funnel and draw filter cake dry. Break up the filter cake thoroughly with the aid of a rubber-policeman, and slurry in the funnel with 50 ml. of 0.5 M nitric acid. Drain and slurry again with 50 ml. of 0.5 M nitric acid and drain. Wash the filter cake with distilled water until a portion of the filter cake, treated with 4 to 5 drops of diphenylamine reagent on the spot plate, does not produce a blue color. About 4 or 5 washings should suffice to give a negative test for nitrates. Draw dry and fluff up the cake with a rubber-policeman. Slurry the cake in the funnel twice with 50 ml. portions of acetone. Fluff up and draw air through the funnel for 2 to 3 minutes. Transfer the cake to a weighing dish and dry in an oven maintained at 105 degrees C. for 1 1/2 hours. Cool in a desiccator. Place an accurately weighed portion of approximately 1 gm. of the dried CMC cake in a 400 ml. beaker. Add 100 ml. of distilled water and 25 ml. of standard N/3 sodium hydroxide, from a burette. Cover the beaker with a watch glass, heat to boiling on a hot plate and boil gently until completely dissolved (10-15 minutes is usually sufficient). Back titrate with standard N/3 hydrochloric acid to a phenolphthalein end point. Apply burette and temperature corrections. Calculate the D.E. as follows:

Equation 1 - Calculation of Factor M

$$M = \frac{(AB) - (CD)}{W}$$

Where:

A = ml. of sodium hydroxide used.
B = normality of sodium hydroxide
C = ml. of hydrochloric acid used
D = normality of hydrochloric acid
W = gms. of dried CMC cake used

Equation 2 - Calculation for determination of D.E.

$$D.E. = \frac{0.162\ M}{1.000 - 0.058M}$$

(NOTE: This is the D.E. value which should meet the requirement).

where:

M = value obtained in Equation 1

4.3.9 Purity-Code Number 14001

4.3.9.1 Determine the purity of the NaCMC in accordance with the method described in ASTM-D-1439-64T.

5. PREPARATION FOR DELIVERY

5.1 Packing

5.1.1 Drums.-Unless otherwise specified in the contract or purchase order, sodium carboxymethyl cellulose shall be packed in non-returnable fiber, plywood or metal drum in accordance with Specification PPP-D-723 (Type III, Grade A), MIL-P-112, and PPP-D-729 (Type III, Full Removable Head Type), respectively. Fiber and plywood drums shall be furnished with a nominal 0.004 inch thick polyethylene liner, properly heat sealed or otherwise closed to afford protection to the contents equivalent to that given by a heat seal closure. (Alternately, an equivalent degree of protection may be furnished by suitable laminations or coatings of the inner surface of the drum.) The drum shall be closed in accordance with Specifications PPP-D-723, PPP-D-729 and MIL-P-112 as applicable.

5.1.1.2 Level C.-Sodium carboxymethyl cellulose shall be packed to afford protection against damage during direct shipment from supply source to the first receiving activity for immediate use. Containers shall comply with Uniform Freight Classification Rules and Container Specifications for rail shipments or National Motor Freight Rules and Container Specifications for truck shipments, as applicable.

5.2 Marking.-In addition to any special marking required by the contract or purchase order, containers shall be marked in accordance with Standard MIL-STD-129. Marking shall include, but not be limited to, the following information:

a. Manufacturer's name
b. Product designation
c. Lot number
d. Date of manufacture
e. The number of this specification

5.3 Storage limitations.-Material shall be stored in a weatherproof storage facility.

6. NOTES

6.1 Ordering data.-Procurement documents should specify the title, number and date of this specification.

6.2 Intended use.-The sodium carboxymethyl cellulose is intended for use as a granulating agent in the manufacture of Lead Azide.

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

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