MIL-B-10854(CmlC)
22 January 1951
SUPERSEDING
Army 96-131-304
12 December 1945

MILITARY SPECIFICATION

BINDER, CELLULOSE NITRATE-CAMPHOR

(For Pyrotechnic Mixtures)

1. SCOPE

1.1 This specification covers one grade of cellulose nitrate binder (cellulose nitrate plasticised with camphor) for use in pyrotechnic mixtures.

2. APPLICABLE SPECIFICATIONS AND OTHER PUBLICATIONS

2.1 Specifications. - The following specifications, of the issue in effect on date of invitation for bids, form a part of this specification:

FEDERAL SPECIFICATION

LLL-B-631 - Boxes: Fiber, Corrugated (For Domestic Shipment).

MILITARY SPECIFICATION

MIL-C-5538 - Cellulose Nitrate.

NAVY DEPARTMENT SPECIFICATION

General Specification for Inspection of Material.

(Army.- Copies of specifications may be obtained from the procuring agency or as directed by that agency. Both the title and identifying number or symbol should be stipulated when requesting copies.)

(Navy.- Copies of Federal, Military, and Navy Department specifications may be obtained upon application to the Bureau of Supplies and Accounts, Navy Department, Washington 25, D. C., except that activities of the Armed Forces should make application to the Commanding Officer, Naval Supply Depot,Scotia 2, N.Y. Both the title and identifying number or symbol should be stipulated when requesting copies.)

(Air Force.- Copies of Federal and Military specifications may be obtained upon application to the Commanding General, Air Development Force, Wright-Patterson Air Force Base, Dayton, Ohio. Both the title and identifying number or symbol should be stipulated when requesting copies.)

2.2 Other publications.- The following publications, of the issue in effect on date of invitation for bids, form a part of this specification:

INTERSTATE COMMERCE COMMISSION PUBLICATION

Regulations for Transportation of Explosives and Other Dangerous Articles, etc.

(Information as to the availability of Interstate Commerce Commissio Regulations for Transportation of Explosives and Other Dangerous Artic etc., may be obtained from the Interstate Commerce Commission, Washing 25, D. C.)

MILITARY STANDARD

MIL-STD-129 - Marking of Shipments.

(Copies of Military standards may be obtained from the same source as the specifications.)

3. REQUIREMENTS

3.1 Form.- The binder may be furnished in the following forms:

a. Flake.
b. Granular.
c. Strip (random cut strips are acceptable).
d. Sheet.

The form desired shall be specified in the invitation for bids (see 6.1).

3.2 Appearance.- The material shall be clean and free from foreign material.

3.3 Chemical requirements.- The material shall conform to the chemical requirements shown in table I.

Table I.- Chemical requirements

Requirement	Maximum	Minimum
Cellulose nitrate (by weight)	82.5%	77.5%
Camphor (by weight)	22.5%	17.5%
Nitrogen content of cellulose nitrate (by wt.)	11.1%	10.8%
Ash (by weight)	0.2%	---
Residual solvent	0.8%	---
Material insoluble in acetone at 24° ± 3°C.	0.2%	---

3.4 Material.

3.4.1 Cellulose nitrate.- The cellulose nitrate shall be newly prepared. Reworked stock shall not be employed.

3.4.2 Camphor.- The camphor shall be suitable in all respects for plasticizing cellulose nitrate.

3.5 Workmanship.- The workmanship shall conform to the best commercial practice covering this class of material.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 Lot.

4.1.1 Batch process.- A lot shall consist of the material produced from no more than one manufacturing batch. (See 6.2.)

4.1.2 Continuous process.- A lot shall consist of the material produced from no more than one work shift with no change in process or materials.

4.2 Sampling.- One, one-pound specimen shall be selected as representative of each lot. The specimen shall be placed in a clean, dry container, and labeled to identify the container and lot represented. The specimen shall be tested according to 4.4.

4.3 Inspection.

4.3.1 Naval purchases. For Naval purchases, the general inspection procedures shall be in accordance with the General Specifications for Inspection of material.

4.3.2 Packing and marking.- The inspector shall inspect the packing and marking of the material for compliance with section 5.

4.4 Tests.

4.4.1 Reagents.- Analytical reagent grade chemicals and water shall be used throughout the tests. Blank determinations shall be run in parallel with each test, using the same quantities of reagents used in the test, and corrections shall be applied when significant.

4.4.2 Form.- Visually examine the material during the sampling operation for compliance with the form and appearance requirements.

4.4.3 Cellulose nitrate.

4.4.3.1 Precipitation.- Weigh, to the nearest mg., approximately 2.5 gm. of the specimen into a tared 100-ml. beaker containing a small, tared, glass stirring rod. (If the material is in sheet or strip form it should be cut into square pieces of about 1/4 inch on a side.) Add about 50 ml. of acetone, cover and let stand, with occasional stirring until the specimen softens. Stir well and add sufficient acetone to make a solution which will flow in a fine thread-like stream from the stirring rod. Let stand,stir thoroughly every 2 hours until the solution is free from lumps. The solution should be made as dilute as possible while still retaining the property of flowing in a thread-like stream without breaking into drops. Hold the beaker and rod approximately 5 inches above the surface of 1500 ml. of benzene contained in a 2-liter beaker. Pour the solution into the benzene by tilting the small beaker just sufficiently to maintain a fine thread-like stream. Stir the benzene slowly and continuously with a large stirring rod, winding the precipitated cellulose nitrate thread around the rod to form a soft, fluffy cocoon. Allow the cocoon to remain in the benzene for 20 minutes to harden, then carefully tear to pieces with the fingers. Stir well in the benzene and let stand overnight.

4.4.3.2 Determination of corrected weight of specimen.- Dry the small beaker and rod to constant weight at 100° ± 2°C., cool in desiccator and weigh. Calculate the weight of the specimen used in the cellulose nitrate determination, corrected for loss of residual solvent, as follows:

$$\text{Weight of specimen, corrected} = \frac{A-(B + (B - C) R)}{100}$$

Where: A = weight of small beaker, rod, and specimen
B = weight of small beaker, rod and dried residue
C = weight of small beaker and rod
R = percent residual solvent/100 (see 4.4.5)

4.4.3.3 Determination of cellulose nitrate.- Dry an "ashless" filter paper to constant weight (± 3.5 mg.) in a weighing bottle. Record the weight of the filter paper. Decant the benzene solution (see 4.4.3.1) through the paper, filter the pieces of the cellulose nitrate cocoon on to the paper, transferring particles adhering to the beaker by means of a spatula and a jet of benzene from a wash bottle. Wash the precipitated cellulose nitrate with benzene and drain well. Place paper and precipitate in a 250-ml. beaker and drive off excess benzene on a water bath, then dry for 2 hours in an oven at 100° ± 2°C. Transfer paper and precipitate to a tared weighing bottle, dry in an oven for 20 minutes, cool in a desiccator and weigh. Repeat the drying and weighing until successive weighings do not differ by more than 10 mg. (Avoid unnecessarily long heating as cellulose nitrate slowly loses weight at 100°C. due to loss of nitrogen.) The weight of the precipitate is computed as follows:

$$P = W - (F + B) = N + A$$

Where: W = weight of precipitate, paper, and bottle
F = weight of filter paper
B = weight of weighing bottle
N = weight of precipitated cellulose nitrate
A = weight of ash
P = weight of the precipitate.

Transfer the precipitate and paper to a tared platinum dish. Pour a known weight of melted paraffin, of known ash content, over the precipitate, burn gently and weigh. The percent ash is computed as follows:

$$\text{Percent ash} = \frac{M \times 100}{S}$$

Where: M = weight of ash corrected for ash of paraffin used
S = weight of specimen, corrected (see 4.4.3.2).

Calculate the cellulose nitrate content as follows:

$$\text{Percent cellulose nitrate} = \frac{(P - A) \times 100}{S}$$

Where: P = weight of precipitate
A = weight of ash
S = weight of specimen, corrected (see 4.4.3.2)

4.4.4 Nitrogen content of cellulose nitrate.- Prepare and weigh, to the nearest mg., approximately 1 gm. of the purified cellulose nitrate as described in 4.4.3.1 and 4.4.3.3 and determine the percent of nitrogen as described in Specification MIL-C-5538. Correct the weight of the specimen for the ash content as determined in 4.4.3.3.

4.4.5 Residual solvent.- Weigh, to the nearest cg., approximately 10 gm. of the specimen (cut into small pieces of the material, in sheet or strip form) taking care not to overheat the material, into a tared metal dish with a tight-fitting cover. Heat the dish (uncovered) and contents in an oven at 100° ± 2°C. for 3 hours. Cover, cool in a desiccator, and weigh. Calculate the percent residual solvent in the following manner:

$$\text{Percent of residual solvent} = \frac{\text{loss in weight} \times 0.6^* \times 100}{\text{weight of specimen}}$$

*0.6 is a factor to compensate for loss of camphor.

4.4.6 Camphor.- Weigh, to the nearest mg., approximately 5 gm. of the specimen. (If the material is in the form of sheet or strip, cut the specimen into small pieces.) Place the weighed specimen in 500-ml flask and add approximately 200 ml. of water and 2.0 to 3.5 gm. of sol: potassium hydroxide. Connect the flask to a condenser and receiver. Heat the flask to decompose the plastic and distill the camphor. It may be found expedient to introduce steam and conduct a steam distillation. Continue the distillation until the camphor is completely distilled. Transfer the contents of the receiver to a separatory funnel. Wash the receiver with three separate 10-ml. portions of benzene, and use the washings to transfer the camphor from the condenser into the separatory funnel. Shake the contents of the funnel and separate the benzene solution. Wash the condenser with three additional 10-ml. portions of benzene and add them to the aqueous distillate and extract a second time. Separate the benzene extract and add it to the first extract. Wash the receiver with two final 10-ml. portions of benzene and use them for a third extraction of the aqueous distillate. Separate the benzene fraction and add to the benzene extracts previously obtained. Dry the combined benzene extracts with anhydrous copper sulfate. Remove the copper sulfate by filtration and wash the copper sulfate on the filter with a small quantity of dry benzene, catching the filtrate and washings in a 100-ml. volumetric flask. Make up to the mark with benzene and determine the camphor content cryoscopically using a Beckmann thermometer. Compare the result with data obtained with known quantities of camphor in benzene solution. Calculate the percentage of camphor in the specimen.

4.4.7 Ash (total).- Weigh, to the nearest mg., approximately 5 gm. of the specimen (cut into small pieces for sheet or strip) and transfer to a tared silica or platinum dish. Add a small amount of nitric acid and digest on a steam bath. Ignite to complete combustion at a low red heat, taking care to prevent loss of ash by air currents. Cool in a desiccator, weigh to the nearest mg., and calculate percent of ash.

4.4.8 Material insoluble in acetone.- Weigh, to the nearest mg., approximately 2 gm of the specimen and dissolve in 32 ml of acetone at 24° ± 3°C. If the solution is clear it is considered that the specimen complies with the requirements of solubility. If the solution is not clear, filter on a tared Gooch crucible. Wash with 5 ml. of acetone. Dry for 1 hour in an oven at 100 ± 2°C., cool in a desiccator, and weigh to the nearest 0.1 mg. Calculate the percentage of insoluble matter.

4.5 Rejection and resubmission.- If the specimen selected as specified in 4.2 fails to conform to this specification, the lot shall be rejected. The contractor, at no expense to the Government, shall have the option of having an analysis made on each container in the lot and removing the nonconforming material, and resubmitting the remaining portion of the lot for acceptance testing. If the specimen taken from the resubmitted portion of the lot fails to conform to this specification, the lot shall be finally rejected.

5. PREPARATION FOR DELIVERY

5.1 Packaging and packing

5.1.1 Sheets.- The sheets shall be furnished in uniform flat packages (or rolls) of approximately 25 or 50 pounds. The packaged sheets (or rolls) shall be wrapped and packaged in fiberboard containers conforming to Specification LLL-B-631. The shipping containers shall be sealed.

5.1.2 Strips.- The strips shall be packaged uniformly in approximately 25 or 50 pound packages. The packaged strips shall be packed in shipping containers as required for sheets.

5.1.3 Granules.- The granular material shall be packaged uniformly in fiberboard drums conforming to Specification No. 21A of the Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc., containing approximately 50 or 100 pounds net weight. The filled drums shall be sealed.

5.1.4 Flakes and random cut strips.- The flake and random cut material shall be packaged in the manner specified for the granular material (see 5.1.3).

5.1.5 Shipping containers. All shipping containers shall afford adequate protection against damage (including moisture addition) and loss of contents, during shipment and storage.

5.2 Marking.- In addition to any special marking required by the contract or order, all shipping containers shall be marked in accordance with the Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc. and Standard MIL-STD-129.

6. NOTES

6.1 Ordering data.- Procurement documents should specify the following:

a. Title, number, and date of specification.
b. Form of material required (see 3.1)

6.2 Batch.- A batch is defined as that quantity of material which has been subjected to some unit chemical or physical mixing process intended to make the final product substantially uniform.

6.3 Inspection.- Inspection will not necessarily include all specification requirements; however, the omission of inspection does not constitute a waiver of these requirements.

6.4 Sampling and testing.- If the contractor consistently produces high-quality material and operates under a system of quality control acceptable to the Government, the Government, at its discretion, may modify, in whole or in part, the sampling and testing procedures specified herein. However, the Government reserves the right to return at any time, without previous notice to the contracto to the sampling and testing procedures specified in this specificatio

Custodian:

Army - Chemical Corps.

Other interest: