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#### MILITARY SPECIFICATION

# PROPELLANT, CANNON, NACO - (U)

- 1. SCOPE
- 1.1 Scope This specification overs "cool" non-picrite propellants (NACO) 1/ suitable for use in cannon.
- 1.2 Classification Propellant shall be furnished in one of the following types compositions and classes, as specified in the applicable requisition, contract or order.
- 1.2.1 Types The classification of propellants, under this specification with respect to the chemical formulation (flame temperature level) is designated by types as follows. The term "Flame temperature" as used in this specification indicates the adiabatic flame temperature calculated from the chemical composition.
  - Type I Propellant of 2000°K nominal flame temperature
  - Type II Propellant of 2200°K nominal flame temperature
  - Type III Propellant of 2150°K nominal flame temperature
- 1.2.2 Compositions Designation of composition is as follows:
  - Composition A Propellants containing ethyl centralite (EC) as a coclant but no potassium sulfate flash-suppressant (EC-NACO)
  - Composition B Propellant containing ethyl centralite (EC) as a coolant and potassium sulfate as a flash-suppressant (EC-NACO)

FSC 1375

- Composition C Propellants containing butyl stearate (BS) as a coolant but no potassium sulfate flash-suppressant (BS-NACO)
- Composition D Propellants containing butyl stearate (BS) as a coolant and potassium sulfate as a flash-suppressant (BS-NACO)
- 1 The Term "NACO" designates the Navy "cool" single-base propellant types which contain low-nitration nitrocellulose (12.0% N, nominal).
- 1.2.3 Classes Designation of grain form is as follows:
  - Class 1 Cylindrical multi-perforated grains
  - Class 2 Cylindrical single-perforated grains
- 2. APPLICABLE DOCUMENTS
- 2.1 The following specifications, standards and publications of the issue im effect on the date of invitation for bids, form a part of this specification.

## SPECIFICATIONS

### MILITARY

JAN-P-193	Potassium Sulfate (for Ordnance use)
JAN-E-199	Ether, Diethyl
MIL-P-231	Propellant, Pyrocellulose
Jan-n-244	Mitrocellulose (for use in Explosives)
Jan-e-255	Ethyl Centralite (Carbamite)
JAN-P-270	Powder, Propellant, Cannon
JAN-P-309	Powder, Propellant, Cannon
MIL-E-463	Ethyl, Alcohol (For Ordnance use)
MIL-L-18618	Lead Carbonate, Basic, Dry (for Ordnance use)
MIL-B-21465	Butyl, Stearate - mormal

#### STANDARDS

#### MILITARY

MIL-STD-129 Marking for Shipment and Storage.

#### **PUBLICATIONS**

#### NAVY DEPARTMENT

#### BURRAU OF ORDNANCE

- OP 5 Ammunition Ashore Handling, Stowing and Shipping
- OP 400 General Specifications for the Manufacture and Inspection of Ordnance Material for U.S. Navy
  - O.S. 1666 Powder, Propellant, U.S. Navy Guns (Ballistic Appendix)
  - MR 10h Revision of Additive Constants for The Simple Calculation of Thermochemical Properties for use in Ballistics (Naval Powder Factory Memorandum Report)

(Copies of specifications, standards, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring agency or as directed by the contracting officer.)

2.2 Other Publications - The following publication, of the issue in effect on date or invitation for bids, forms a part of this specifications:

49 CFR 71-78 - Code of Federal Regulations

(The Interstate Commerce Commission regulations are now a part of the Code of Federal Regulations (1949 Edition-Revised 1950) available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Orders for the above publication should cite "49 CFR 71-78 (rev. 1950)".)

- 3. REQUIREMENTS
- 3.1 Materials
- 3.1.1 Raw-Materials The raw materials used in the manufacture of the propellant shall conform to the corresponding specifications listed in 2.1, and in addition, the following materials shall be of the type, grade, of class specified hereafter.

- 3.1.1.1 Nitrocellulose Nitrocellulose, grade E, of Specification JAN-N-244 containing 12.00 f.10 percent nitrogen, with a viscosity of 5 f 2 seconds and a fine-ness of 96 f 6 ml., made from cotton cellulose shall be used. The required nitrogen content may be obtained by blending nitrocellulose containing from 11.90 to 12.20 percent nitrogen. Nitrocellulose made from wood cellulose or nitrocellulose recovered from scrap powder shall not be used without prior approval of the contracting agency.
- 3.1.1.2 Ethyl Alcohol Grade 2 of Specification MIL-E -463.
- 3.1.1.3 Ether, Diethyl Grade A of Specification JAN-E-199.
- 3.1.1.4 Ethyl Centralite Class 2 of Specification JAN-E-255.
- 3.1.1.5 Potassium Sulfate Grade A of Specification JAN-P-193.
- 3.1.1.6 Butyl Stearate Specification MIL-B-21465
- 3.1.2 Reworked Materials
- 3.1.2.1 Normal Scrap Normal scrap colloid from mixers, blocking presses, extruding presses, cutting machines and other units shall be collected frequently into covered receptacles, and kept soft with solvents. This colloid may be used in the mixers, either by itself, or mixed with fresh nitrocellulose. It shall be further softened, if necessary, in the mixer.
- 3.1.2.2 Hard Scrap— Hard colloid or hard rework from any operation of the same composition as the material in process, or base grain accumulating between solvent recovery and coating operation, if clean and not damaged by heat or sunlight, may also be softened with solvents and used with fresh nitrocellulose in mixing. The quantity shall be such as to produce a uniform colloid, but shall usually be not over 10 percent of the mixer charge.
- 3.2 Composition
- 3.2.1 Nominal Formulation The product shall contain, in approximately the proportions by weight shown in Table I, or Table II as appropriate,

all of the ingredients specified for each propellant type, as indicated. The general control of propellant composition under this specification is by means of the test and performance requirements set forth, particularly the gun ballistic requirements, and the closed bomb relative force which, for a given propellant type, is proportional to the flame temperature. (The chemical analysis shall be reported on the description of each powder lot.)

Table I. Nominal Chemical Composition, EC-NACO

Constituent	Percentages by Weight			
	Type I Comp A	Type I Comp B	Type II Comp A	Type II Comp B
Nitrocellulose	89.0	87.5	91.4	89.4
Ethyl Centralite	6.0	6.0	3.8	2.8
Basic Lead Carbonate	1.0	1.0	1.0	1.0
Potassium Sulfate		2.0		3.0
Total Volatiles	4.0	_3.5	_3.8	3.8
	100.0	100.0	100.0	100.0

Table II. Nominal Chemical Composition, BS-NACO

Constituent	Percentages by Weight			
,	Type I Comp C	Type I Comp D	Type III Comp C	Type III Comp D
Nitrocellulose .	90.6	89.2	91.7	89.3
Butyl Stearate	5.9	5.3	3.3	2.7
Ethyl Centralite	1.0	1.0	1.0	1.0
Basic Lead Carbonate	1.0	1.0	1.0	1.0
Potassium Sulfate	***	2.0		3.0
Total Volatiles	1.5	1.5 -	3.0	3.0

3.2.2 <u>Flame Temperature</u> - The calculated flame temperature of each propellant lot shall be reported for information. The calculated flame temperature of Type I propellant lots shall not exceed 2050°K.

- 3.2.3 Ethyl Centralite Any type of propellant covered by this specification shall contain a minimum of 1.0% ethyl centralite to act as stabilizer. (The nominal formulations listed in Table I indicate approximately total percentages of ethyl centralite necessary in a propellant meeting performance and test requirements.)
- 3.2.4 Basic Lead Carbonate The percentage of this anti-fouling agent shall be within the range 1.0 \( \frac{1}{2} \) 0.2%.
- 3.2.5 Potassium Sulfate For the propellant types containing potassium sulfate as flash-suppressant, the content shall be that specified within a tolerance range of \$\notineq 0.3\% K\_2SO\_4\$. The requisition, contract, or order may specify other percentages of flash-suppressant than those listed in Table I.
- 3.3 Grain Form and Dimensions The grain form and dimensions shall comply with the requirements listed in JAN-P-270. Waiver of the JAN-P-270 specification may be secured by approval of the Bureau of Ordnance if necessary to satisfy PPD (Production Packing Department) provided the ballistic requirements are satisfactory.
- 3.3.1 Web The web of all propellant lots shall be of such magnitude that the propellant meets the ballistic requirements, after having met the flame temperature and volatile content requirements of sections 3.2.2 and 3.2.1 (Note: It is recommended that Type I propellant lots be controlled in the drying by use of closed bomb and total volatiles determination; however, such controls are left to the discretion of the mammifacturer.)

#### 3.4 Test Regulremente.

- 3.4.1 Density The minimum permissible value shall be 1.55 grams/cc.

  The preferred minimum density value is 1.57. (Attention is called to the fact that density is a factor affecting the packing characteristics; all propellant lots must fit, with satisfactory packing depth, into the cartridge case for the applicable gun.)
- 3.4.2 Stability When subjected to the 134.5°C heat test, the propellant shall not cause complete fading of the methyl violet test paper to a salmon pink color in less than one hour. Explosion shall not occur in less than five hours.

### 3.4.3 Closed Bomb Requirements

- 3.4.3.1 Relative Force The measurement of relative force shall be made in a closed bomb. The reference propellant shall be supplied by the Bureau of Ordnance and shall be representative of nominal composition of the propellant type to be tested, preferably the standard propellant for ballistic acceptance tests. The average relative force (R.F.) value shall be within £2.0% of the standard of reference lot. If the reference propellant is not representative of the desired flame temperature, then the relative force requirement may be changed to cover a range of 4% not centered about the reference propellant.
- 3.4.3.2 Relative Quickness The average relative quickness (R.Q.) value found in the closed bomb test shall be reported for information; there are no specification requirements of R.Q.
- 3.5 Gun Ballistic Requirements The propellant shall comply with the ballistic requirements of Ordnance Specification 1666. Additional requirements as to minimum production packing depth in the cartridge case may be specified in the contract.

### 3.6 Processing

3.6.1 Approval of Equipment and Processes - Details of the manufacturing

Details of the manufacturing process, and description of the equipment used by the contractor shall be submitted to the Chief of Bureau of Ordnance, Department of the Navy, Washington, D.C., in writing and written approval received, prior to commencement of manufacture. Any deviation from the approved manufacturing process must be submitted in writing and receive the written approval of the Chief of Bureau of Ordnance, Department of the Navy, Washington, D.C., prior to use.

- 3.6.2 Recommended Manufacturing Processes The following procedures have been successfully used for the manufacture of NACO propellants.
- 3.6.2.1 Dehydration of Nitrocellulose The nitrocellulose is dehydrated with ethyl alcohol, to remove all water.

  At least 1.0 pound of alcohol for every pound of dry nitrocellulose in the press charge is pumped through the block, including the alcohol left in the block, having no more than is required for mixing. (Approximately 23%).
- 3.6.2.2 <u>Block Breaking</u> The blocks of dehydrated nitrocellulose are Broken up in a suitable block breaker, equipped with screen to remove all lumps and the mixer charge is weighed.
- 3.6.2.3 Mixing The weighed charge of nitrocellulose is placed in a suitable mechanical mixer in which the blade action may be reversed, and is thoroughly agitated for ten minutes and sampled for T.V. The required amount of basic Lead Carbonate (Type I-A, II-A) or of basic Lead Carbonate and Potassium Sulfate (Type I-B, II-B) are then added and dry mixed for about ten minutes when the required amounts of alcohol, ether, and ethyl centralite dissolved in the ether are added, and the charge thoroughly mixed for one hour, reversing the blade action several times to insure proper incorporation of the ingredients. The solvent for this type nitrocellulose should consist of 75-80 percent alcohol and 20-25 percent ether and the solvent ration compared to weight of dry ingredients should be between approximately 0.9 and 1.2 depending upon the discretion of the manufacturer and the extent of solvent losses during mixing. However, the exact solvent mixture, the amount of solvent and the mixing time shall be sufficient to give thorough colloiding and distribution of all ingredients. For propellants of the compositions C and D, the weighed charge of nitrocellulose is placed in a suitable mechanical mixer in which the blade action may be reversed. The required amount of basic lead carbonate (Type IC and IIIC) or of basic lead carbonate and potassium sulfate (Type ID and IIID) is added and dry mixed for five (5) minutes. The required amount of butyl stearate and alcohol are mixed and added to the mixer. The required amount of ethyl centralite is then dissolved in the required amount of other and added to the mixer. The charge is then thoroughly mixed for at least 25 minutes,

reversing the blade action several times to ensue proper incorporation of the ingredients. The solvent for this type nitrocellulose should consist of 75-80 percent alcohol and 20-25 percent either and the solvent ratio compared to weight of dry ingredients should be between approximately 0.8 and 1.0 depending on the discretion of the manufacturer and the extent of solvent losses during mixing. However, the exact solvent mixture, the amount of solvent and the mixing time shall be sufficient to give thorough colloiding and distribution of all ingredients.

3.6.2.4 Pressing - The charge from the mixer shall be blocked for sufficient time and at a pressure which has been previously determined to give thorough consolidation. At the discretion of the manufacturer, the colloid may them be strained in a "macaroni" press in order to promote more thorough colloiding and to remove uncolloided nitrocellulose, and foreign particles such as pieces of metal and wood. If colloid is not strained in a macaroni press, screens must be used in the finish press. The colloid shall then be blocked and pressed through dies to form strands. The strands shall be granulated by passing through cutting machines into grains of such dimensions that after proper drying shall have the desired final dimensions and perforations. The screens used shall be such as to reduce to a minimum the amount of uncolloided nitrocellulose in the finished grains. Unless found free of foreign particles, the material left on the screens shall not be reworked.

NOTE: - At the discretion of the manufacturer, the colloid may be strained more than once in the "macaroni" press to produce a better colloid and one that will work better in the finishing presses.

3.6.2.5 Solvent Recovery - The powder shall be subject to a solvent recovery process having for its object both the gradual removal from the powder grains of the bulk of, and the economic recovery of the volatile solvents, alcohol and ether, used in their manufacture. The rate at which the temperature is increased shall be left to the discretion of the manufacturer, the rate being such as to prevent undue distortion of the powder grains. The temperature at any time during the solvent recovery process shall not exceed 55°C. The solvent recovery process shall be continued until the grains are shrunk down to the extent that further processing, such as

water-drying or air-drying will not result in additional distortion of the powder grains.

- 3.6.2.6 Water-Drying or Air-Drying After being shrunk down and freed of the bulk of its volatile solvents during the solvent recovery process, the powder shall be dried in air at a temperature not exceeding 55°C or steeped in water at a temperature not exceeding 55°C for a time sufficient to reduce the volatile solvent content to the desired value. The number of changes of water during the steeping of the powder shall be left to the discretion of the manufacturer. At his discretion, the manufacturer may subject the powder, after the solvent recovery treatment. to an air-drying treatment at a temperature not exceeding 55°C in order to reduce the volatile solvent content before steeping the powder in water and thereby improve the appearance of the finished powder. After the volatile solvent content of the powder has been reduced by the watersteeping process, the powder shall be removed from the water, and the excess water removed by draining followed by air-drying at a temperature not to exceed 55°C. The propellants should be packed within 24 hours after completion of the drying cycle or after the heat is turned off. If it is impossible to pack powder at the completion of drying within the stated time then the powder should be subjected to a 24-hour drying period just prior to packing. These precautions are necessary because of the somewhat hygroscopic nature of these propellants.
- 3.6.2.7 Blending Propellants of compositions C and D, on removal from the dry house, should not be unnecessarily exposed to atmospheric conditions. The propellant should be unformly blended in lots of prescribed size.
- 3.6.2.8 <u>Handling</u> The propellant and its standard ingredients shall at all times be protected from the action of direct sunlight and acid fumes.
- 3.6.2.9 <u>Sorting</u> The propellant shall be sorted thoroughly so as to remove cracked, distorted, short and long grains and otherwise deformed grains.
- 4. QUALITY ASSURANCE PROVISIONS AND TEST REQUIREMENTS.
- 4.1 Lot Unless otherwise specified, NACO propellants shall be manufactured in lots of 50,000 pounds minimum and 550,000 pounds, maximum.

- 4.2 <u>Sampling</u> The amount of propellant required to make up the ballistic, chemical and stability and closed bomb samples shall be taken from containers selected so as to be representative of the lot.
- 4.2.1 Ballistic Samples The weight of the ballistic sample shall be as specified in 0.3. 1666. If the weight of the samples required is less than 7 full containers, a portion equal to one-seventh of the weight of the required ballistic sample shall be removed from each of 7 boxes. These 7 portions shall be packed in individual airtight containers. The ballistic sample may be taken as soon after packing as desired, except when moisture has been added to the propellant while in the blending tower, in which case the sample shall be taken sooner than 48 hours after the time of packing.
- 4.2.2 Chemical and Stability Samples From each of the containers asmpled for the ballistic tests, an equal portion of propellant shall be taken, so as to have a total weight for the chemical and stability tests as specified in 0.3. 1666. Portions of these original samples shall be set aside for the 134.5 C heat test specified in paragraph 4.2.3 and the balance blended thoroughly to form a composite chemical sample. Five pounds of the composite chemical sample shall be taken for the 65.5 C surveillance test (run for information only) and forwarded to the Supply Officer, Naval Propellant Plant, Indian Head, Maryland. The greatest possible cleanliness shall be observed in handling the chemical and stability samples, and touching them with damp or soiled hands shall be avoided.
- 4.2.3 Packing and Marking of Samples All samples shall be packed in airtight containers, preferably in glass jars if the samples are small, or in propellant boxes which have been tested and found airtight immediately before use.

  Each sample container shall be marked to show the propellant designation, lot number, manufacturer, contract number, number of pounds in the lot, and the number of the box from which the sample was taken.
- 4.2.4 Closed Bomb Sample The amount of propellant required to make up the closed bomb sample as specified below shall be taken from containers selected so as to be representative of the lot. A portion equal to one-seventh of the weight of the required closed bomb sample shall be removed from each of seven boxes and these seven portions shall be blended and

packed in an air-tight container.

Web Size

Closed Bomb Sample

0.10 inch or less above 0.10 inch 7.1b

- 4.3 <u>Inspection and Test Procedures.</u>
- 4.3.1 Grain Form and Dimensions. The procedure described in MIL-P-231 shall be used.
- 4.3.2 Density.
- 4.3.2.1 Apparatus. Vacuum pump, with a rated gauge pressure of 5 mm or less. Specific Gravity Bottle, 50 ml, equipped with a thermometer and side arm cap.
- 4.3.2.2 Calibration of Specific Gravity Bottle. Dry the pycnometer carefully. (Do not dry the thermometer in an oven..) Weigh assembly to the nearest milligram. Fill with freshly boiled distilled water that has been cooled to 20-21°C. Insert the thermometer in such a manner as to expel all air from the pycnometer. Adjust the temperature to 25.0°C. Wipe the side arm dry with a towel and place on cap. Wipe the assembly several times with a clean dry towel and weigh.
- 4.3.2.3 <u>Displacement Liquid</u>. Prepare solution by dissolving approximately 0.05 gm of aerosol OT in one liter of freshly boiled distilled water. Cool to about 20°C. Fill the pycnometer, and adjust the temperature to 25°C and weigh, as under 4.3.2.2 "Calibration".

Density (gm/ml) of displacement liquid

where B = weight of displacement liquid

A = weight of water

D = density of water @ 25°C

4.3.2.4 Procedure. - Prepare, with a minimum of cutting, a representative portion of the sample in pieces that will pass the neck of the pycnometer. Weigh approximately 10 grams of sample to \$\neq\$1 mg and transfer them to the pycnometer. Add enough of the

aerosol water solution to cover the sample. Place the pycnometer in a vacuum oven (or other shielded container) and evacuate to 5mm of mercury for 15 minutes. Release the vacuum. Chill the pycnometer and contents to approximately 2000. Fill the pycnometer, insert the thermometer, adjust the temperature of the pycnometer to 25°C, cap the side arm, dry the pycnometer thoroughly and weigh to the nearest milligram.

Density = 
$$\frac{WD}{(P1 \neq W) - (P_2)}$$

where W = weight of sample

d = density of displacement liquid
P1 = weight of pycnometer when filled

P<sub>1</sub> = weight of pycnometer when filled with the displacement liquid

P<sub>2</sub> = weight of pycnometer when filled with sample and displacement liquid

- 4.3.3 Stability. The 134.5°C heat test shall be made as directed in JAN-P-270.
- 4.3.4 Closed Bomb Test. The following procedures are to be used for obtaining relative force and relative quickness data on NACO cannon powder. The relative force (R.E.) shall represent the average of two tests each consisting of three shots fired in a closed bomb. The relative quickness (R.Q.) shall be calculated and included in the report for information. There shall be no deviations from this procedure without prior approval of the Bureau of Ordnance.
- 4.3.4.1 Apparatus. The apparatus shall consist of a closed bomb of appropriate size, a piezo electric gage, cathode ray oscillograph and associated electrical apparatus, and a recording camera all in accordance with the following description, and of a type approved by the Bureau of Ordnance.

4.3.4.1.1 Closed Bombs. - Each closed bomb shall consist of an alloy steel cylinder, water jacketed for temperature control, and provided with an electrically operated firing head, a gage head designed to house a piston and a piezp-electric gage, and a needle valve for releasing the gaseous products of explosion. The bombs used will be of a design approved by the Bureau of Ordnance. The following table gives the drawing number of approved bombs and the size of bomb which should be used in the tests.

Model No.	Volume	DuPont Drawing No.	Powder Size
6	200	01635, 01636, 01656	op to 106 M. W.
12 7 13	700	01700 01642, 01653 01700	706 to 110

The web will be determined by the nominal web of the granulation manufactured.

- Piezo-electric Gage. The piezo-electric gage shall consist of a number of x-cut quartz plates assembled in such a way that the pressure developed by the combustion of a powder charge in the closed bomb may be applied normally to the faces of the plates, and provided with electrical connections permitting the collection of the electrical charges of both signs produced by compression of the gage. A gage of suitable design is described in DuPont Co., Burnside Laboratory Detail No. 01550. The gage may be calibrated by applying or releasing known loads and measuring by suitable apparatus the quantity of electricity produced.
- 4.3.4.1.3 Cathode ray oscillograph. The cathode ray oscillograph shall be so constructed as to transform the electrical impulse produced by the piezo-electric gage during combustion of the powder into a visible trace capable of being photographed. Horizontal deflection of the luminous spot on the face of the oscillograph shall be substantially proportional to pressure in the bomb at any instant, while vertical deflection shall be substan-

tially proportional to rate of pressure rise at the same instant. The oscillograph shall be provided with calibrating apparatus such that horizontal and vertical deflection of the luminous spot can be related accurately to input voltages. Calibration shall consist of the successive formation of luminous spots on the face of the cathode ray tube at known input voltage intervals so as to form a system of essentially rectangular coordinates on the tube face capable of being photographed.

4.3.4.1.4 Recording Camera. - The camera shall be such as to permit the operation to obtain a permanent photographic record of the luminous trace produced on the face of the cathode ray tube by the combustion of a powder charge in the bomb together with the calibration pattern.

#### 4.3.4.2 Test Procedure.

#### 4.3.4.2.1 Preparation of Charges.

4.3.4.2.1.1 Bomb Charge. - The bomb charge for NACO formula cannon powder shall consist of sufficient powder and igniter to give a loading density of 0.250. In the calculation of loading density, the following smokeless powder equivalent shall be used:

Nitrocotton 1.00 Igniter or blank 1.00 Fire powder

The powder charge shall be taken from a blend of samples and shall be weighed to a constant weight and shall be accurate to 0.5%. All powder grains shall be whole with the exception of the one used to adjust the charge to the correct weight.

4.3.4.2.1.2 <u>Ignition Charge.</u> - The ignition charge shall consist of .10 grams of dry unpulped but stabilized High Grade Nitrocellulose and the following amounts of a suitable smokeless igniter (or blankfire powder, e.g. EC, S.R. 4907, or S.R. 4990) or equivalent.

Bomb Size	Black Powder	Smokeless igniter
200 cc 700 of 800 cc		1.0 grams 5.0 grams

- 4.3.4.2.1.3 Powder temperature. The powder charges both test and standard, shall be within plus or minus 2°F of the same temperature before firing and shall be exposed to the atmosphere a minimum length of time during preparation.
- Bomb temperature. The temperature of the inside of the bomb shall be held constant (plus or minus 10°F) during the test. One warming round shall be fired before the test to bring the bomb up to temperature.
- 4.3.4.2.3 <u>Test firings.</u>
- Number of Shots to be fired, Three shots each of the lot or lots under test, as many as three lots may be fired at one time, and the standard powder shall be fired in alternation: i.e., lst round standard, lst round lst lot, lst round 2nd lot, lst round 3rd lot, 2nd round standard, 2nd round lst lot, etc. Abnormal records for which the cause is established may be replaced if detected during the firing so that there is a minimum of three good records for each powder. If the quickness, as defined below, of the three shots has an extreme variation greater than 3.0% the test shall be repeated.
- 4.3.4.2.3.2 Quickness. The relative quickness and relative force of the lot under test shall be determined by taking the average values of two tests as described in 4.3.4.2.3.1. The relative quickness and relative force of the two tests shall not vary by more than two percent. If the difference between the two tests exceeds this limit, a third test shall be fired and the relative quickness and force reported for the lot shall be the average of all three tests unless one test is obviously incorrect.
- 4.3.4.2.4 <u>Standard powder.</u> The standard powder shall be the same standard as that specified by the Bureau of Ordnance to be used in gun firing acceptance of the granulation under test.
- 4.3.4.2.5 <u>Circuit constants.</u> The condenser in the pressure circuit and the resistance in the burning rate circuit shall be of such values that the burning curve is the convenient maximum size that can be wholly contained within the grid of calibration points. No change shall be made in any of the circuit constants during a test.

## 4.3.4.2.6 Measurement of Records.

- 4.3.4.2.6.1 Determination of Relative Quickness. Measure the rate of pressure rise in volts, of each record at each of four pressures corresponding to 0.50, 0.75, 1.00 and 1.25 volts horizontal deflection or at other convenient voltages in like proportions. The average rate of pressure rise of the standard powder at the same pressure and multiplied by 100 is the relative quickness of the lot at that pressure. The average value of this figure for the four pressures shall be the relative quickness of the lot referred to the standard powder, the quickness of which is taken as 100.
- 4.3.4.2.6.2 Determination of Relative Force. Maximum horizontal deflection on the oscillograph record of a bomb firing is a measure of the maximum pressure produced during the explosion. At constant loading density, maximum pressure is proportional to force of the powder.

  Measure the maximum pressure in volts of the shots fired of the lot under test at the maximum abscissa of the explosion trace. Average these figures. Obtain the average maximum pressure of the standard powder in the same way. The average maximum pressure of the standard expressed in the same units and multiplied by 100 shall be the relative force of the lot referred to the standard powder, the force of which is taken as 100.
- 4.3.5 Gum Ballistic Test. These tests shall be conducted in accordnace with NAVOHD O.S. 1666.
- 4.3.6 Chemical Analysis. Each lot of propellant shall be analyzed by the following procedures, except that the determination of the degree of nitration of the nitrocellulose in the propellant (4.2.6.6) is to be made only when specifically requested.
- 4.3.6.1 Total Volatiles. Hercules Solution Evacuation Method. The procedure described in MIL-P-231 shall be used for the Total Volatiles determination.
- 4.3.6.2 Ethyl Centralite. Active.
- 4.3.6.2.1 Preparation of Sample. Cut, slice, plane, or microtone a representative portion of the sample grains into thin

wafers 0.01 to 0.02 inch thick. It is not advisable to grind the samples in a Wiley Mill or other grinding devices.

- 4.3.6.2.2 Separation of the Ethyl Centralite.
- 4.3.6.2.2.1 Extraction with Methylene Chloride. Place an accurately weighed sample of approximately 5 gm in an extraction thimble and assemble into a Soxhlet extractor. Extract, continuously, with CP methylene chloride on a stream or hot water bath for 16 to 24 hours.
- 4.3.6.2.2.2 Steam Distillation (optional). Place an accurately weighed sample of approximately 5 gm in a l liter distillation flask, add 200 ml of 15% NaOH. Assemble immediately into a steam distillation apparatus. Distill, at a rate of 4-8 ml per minute, approximately 750 ml. Collect the distillate in a l liter separatory funnel. Add 10 gm of NaCl to the distillate and extract with 3-4 separate 50 ml portions of methylene chloride. Collect the methylene chloride extracts in a 500 ml flask.
- Procedure, Evaporate the methylene chloride solution in a gentle stream of dry, filtered air. Avoid prolonged drying. Dissolve the residue in glacial acetic acid, transfer to a 250 ml volumetric flask and dilute to volume with 70% (by volume) acetic acid. Remove; by pipette, an aliquot to contain .05 to .15 gm of ethyl centralite and transfer it to a 250 ml iodine flask; add 10 ml of 1:1 HCl. Chill in an ice bath to 10° 12°C. Add 20 ml of 0.2N KBr-KBrO3 solution. Stopper the flask immediately and swirl for 75 seconds. Add 15 ml of 15% KI solution to the funnel neck of the flask and remove the stopper carefully. Add 20 ml of carbon tetrachloride and titrate with standard 0.1N sodium thiosulfate to a starch end point.

Make a blank determination using all solutions and reagents as for the sample determination.

% Ethyl Centralite = 6.709 N (B-V)

Where B = Bolume of sodium thiosulfate required to titrate blank
V = Volume of sodium thiosulfate

required to titrate sample

N = normality of sodium thiosulfate

W = weight of sample represented

by aliquot

## 4.3.6.2.4 Solutions.

4.3.6.2.4.1 Starch Indicator. - Make a slurry of 5 gm of soluble starch in 100 ml of cold water and pour into 900 ml of boiling water. Add 0.1 gm of mercuric iodide.

Cool before using.

- 4.3.6.2.4.2 Potassium Bromide—Bromate Solution. Dissolve 5.567 gm of KBrO3, 25 gm of KBr and 0.2 gm of K2CO3 in 200 ml of hot water. Cool to room temperature and dilute to one liter. If prepared accurately, this solution may be used to standardize the sodium thiosulfate solution.
- 4.3.6.2.4.3 Standard Sodium Thiosulfate Solution. Dissolve 25 gm of sodium thiosulfate pentahydrate and 0.1 gm of sodium carbonate in one liter of water. Standardize against standard 0.21 KBrO<sub>3</sub> solution of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution by the following procedure:

Pipette 20 ml of the standard oxidizing solution into a 250 ml iodine flask. Add 25 ml of 15% KI and 10 ml of 1:1 HCl. Stopper the flask and let stand for 5 minutes. Titrate with the sodium thiosulfate solution to a starch end point.

All of oxidizing solution X normality of oxidizing solution = ml of sodium thiosulfate X normality of sodium thiosulfate.

- 4.3.6.2.4.4 Potassium Iodide Solution. Dissolve 15 gm of potassium iodide and 0.1 gm of potassium carbonate in 100 ml of water.
- 4.3.6.2.5.5 Standard Potassium Dichromate (O.IN). Dissolve 4.9035 gm of dry, CP potassium dichromate in water and dilute to 1 liter in a volumetric flask.

4.3.6.3 Basic Lead Carbonate. - Place an accurately weighed sample, of 10-20 gm, in a 400 ml beaker. Add 50 ml of lead-free concentrated nitric acid, cover and heat on a steam bath until solution is effected. Dilute with an equal volume of hot water and filter while hot through an asbestos or fritted glass mat. Receive the filtrate in another 400 ml beaker. Add 10 ml of 1:1 H<sub>2</sub>SO<sub>4</sub> to the filtrate and heat on the steam bath. Add isopropyl alcohol dropwise until the reaction subsides and then add an additional 10 ml of the alcohol. Continue heating for 1/2 hour. Dilute with an equal volume of ethyl alcohol and cool. Filter the precipitate on a tared fine porosity fritted porcelain crucible and wash with 1:1 ethyl alcohol. Ignite at 5000-5500C for 1/2 hour. Cool and weigh.

% Basic Lead Carbonate = 85.26W

where W = weight of lead sulfate S = weight of sample

4.3.6.4 Potassium Sulfate. - Place an accurately weighed sample, of 10-20 gm, in a 400 ml beaker. Add 50 ml of sulfate-free concentrated nitric acid. Cover the beaker and heat on the steam bath until solution is effected. Add an equal volume of hot water and filter rapidly through an asbestos or fritted glass mat into a 400 ml beaker. Add 10 ml of concentrated HCL to the filtrate and heat on the steam bath. Add isopropyl alcohol dropwise and then, after the reaction subsides, add 10 ml of the alcohol. Continue heating for 1/2 hour on the steam bath. Dilute to 200 ml and bring to a boil on a hot plate. Add 10 % barium chloride solution, dropwise, until 10 ml has been added to the boiling solution. Digest the precipitate on the steam bath for 1 to 3 hours. Filter while hot on a tared fritted porcelain crucible of fine porosity. Wash the precipitate thoroughly with hot water. Ignite at 700°C for 1/2 hour, cool and weigh.

% Potassium Sulfate = 74.65%

where W = weight of barium sulfate S = weight of sample 4.3.6.5 Nitrocellulose. - Nitrocellulose is determined by difference.

% Nitrocellulose = 100 - sum of percentage of all other constituents

4.3.6.6 Nitrogen in Nitrocellulose. - This determination is to be made only when specifically requested, since the nitration is controlled by, and normally determined under the raw material specifications. Place approximately 1.4 gm of the sliced sample in a 250 ml beaker. Add 110 ml of CP anhydrous aceptone cover with a watch glass and allow to stand overnight under a bell jar. Stir to effect complete solution and allow to stand one hour to settle the insoluble salts. Decant carefully into a 100 ml Goetz centrifuge tube and centrifuge for 1/2 hour at 1500 RPM. Separate the supernatant liquid carefully and place in a 400 ml beaker. Add dropwise, with vigorous stirring, a 2% sodium chloride solution until a permanent precipitate is formed. Add 100 ml of water and heat on the steam bath. Evaporate to dispell the smell of metone. Add 100 ml of water and heat on the steam bath for 2 hours. Filter hot on a fritted glass mat and wash thoroughly with boiling water. Dry in a vacuum oven at 40 C for 4 hours. Transfer an accurately weighed portion of the dried residue to a 30 ml bottle that is fitted with a ground glass stopper. Add 10 ml of cold 95% £ .5% sulfuric acid, stopper and allow to stand in an ice chest to effect solution (2-4 hours). Transfer the solution, carefully, to a nitrometer with an additional 15 ml of 95% sulfuric acid and proceed as in JAN-N-244, "Nitrocellulose," paragraph F-4a - Nitrogen Content.

% Nitrogen (of nitrocellulose) -  $\frac{R}{W}$ 

Where R = nitrometer reading, in % nitrogen per one gram sample (Nitrocellulose reading tube) W = weight of dried separated nitrocellulose taken

4.3.6.7 n-Butyl Stearate. - Place an accurately weighed sample, to contain 0.3-0.5 grams butyl stearate, of the sliced or microtomed propellant in a coarse sintered extraction thimble. Cover the sample with a loose plug of glass wool. Place the thimble and sample in an all glass sohlet extraction apparatus and fill the apparatus with CP methylene cloride or peroxide free ether. Extract the sample for 16-20 hours over a hot water bath or steam

heated hot plate using an extraction rate of approximately 0.5 ml per minute. After the extraction is completed, remove the flask containing the detractable material and evaporate the solvent in a stream of warm dry air. Add, by pipette, 25 ml. of 0.IN alcoholic potassium hydroxide, fit the flask with an air condenser and heat on the steam bath for 1-12 hours. Prepare a blank determination on the solvent and reagents used. Remove the flask from the steam bath and add 25 ml of water through the condenser. Remove the condenser, stopper the flask and cool to room temperature. Titrate the solution with standard 0.05N HCL to a phenol-phthalein end point.

Where: W = weight of sample

B = ml of HCL required to titrate blank

V = ml of HCL required to titrate sample

N = normality of HCL

- 4.3.6.8 Total Moisture (water). The procedure described in specification JAN-P-309 shall be used for total moisture determination when necessary.
- 4.3.7 Flame Temperature. The adiabatic flame temperature is calsulated from the percentages of the ingredients found by chemical
  analysis. The recommended procedure is described in Naval Powder
  Factory Memorandum Report, 1R104. The following table lists the
  recommended molar additive constants per gram of propellant:

Constituent	$\operatorname{cv}_\mathtt{i}$	Ei	Ni
Nitrocellulose*	0.3464 /(0.0065Y)	113.2 -(143.51)	0.04169 /(0.0022Y)
Ethyl Centralite	0.4013	-2882.	0.10434
Ethyl Alcohol	0.6121	-2772.	0.10853
Water (liq)	0.6403	-1552.2	0.05551
Basic Lead Carbonate	.0910	-199.7	0.00387
Potassium Shlfate	•2708	-662.4	- 0.00574
Butyl Stearate	0.5675	-3726.	0.12919

\* Y - 12.0 - 3N

NOTE: The following water contents are assumed for calculation purposes if not experimentally determined. The remainder of the total volatiles is assumed to be ethyl alcohol.

TIPES	<u>3"/50</u>	3"/70	511/54
II A, IIB I A, I B	0.5%	0.7%	0.7% 0.7%
I C, IIIC	0.2%	0.7%	
I D, HID	0.2%		0.7%

- 4.3.8 Retest. Retests shall be conducted in accordance with specification JAE-P-270.
- 4.4 Description Sheets and Test Reports. With every lot of propellant submitted, a descriptive sheet and test report shall be furnished giving a complete history of its manufacture. Unless otherwise specified, eight copies of each description sheet and report shall be submitted to the Bureau of Ordnance, Department of the Navy, Washington 25, D.C. In addition, two copies shall be furnished to each of the following:

Commanding Officer
U.S. Naval Propellant Plant
Indian Head, Maryland

Commander U.S. Naval Proving Ground Dahlgren, Virginia

- 5. PREPARATION FOR DELIVERY
- 5.1 Preservation and Packaging.
- 5.1.1 Level A. In addition to the following, level A packaging shall be in accordance with 49CF R71 78.
- 5.1.1.1 <u>Unit Packaging.</u> Unit packaging shall be in accordance with specification <u>JAN-P-270</u>. Unit package shall consist of a metal box in accordance with BuOrd drawings listed in specification <u>JAN-P-270</u>.
- 5.1.1.2 Intermediate Package. None
- 5.1.2 Levels B and C. Not applicable
- 5.2 Packing. No overpacking shall be required.
- 5.3 Marking.
- 5.3.1 Special Markings. None, unless otherwise specified.
- 5.3.2 Normal Markings. In addition to the markings required by contract or order, unit packages, intermediate packages (when a mand) and shipping containers shall be marked in accordance

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with the requirements of MIL-STD-129 and 49CFR71-78.

### 6. NOTES.

- 6.1 General Safety Precautions. Responsibility for safety in the manufacture and handling of this propellant shall be that of the manufacturer. Guidance in regard to safety precautions may be obtained by reference to Bureau of Ordnance Pamphlet O.P. 5, "Ammunition Ashore".
- 6.2 <u>Use.</u> The smokeless powder covered by this specification is intended for use as a propelling charge for cannon.
- 6.3 In case the propellant fails to pass the ballistic tests in the weapon for which it was intended, the Bureau or agency concerned may, if deemed to its interest, accept the powder, upon its meeting the specifications of the Bureau or agency concerned for any other weapon.
- 6.4 Loading of Ammunition. Strict adherence to the specified loading conditions given in OP 5 as to temperature and humidity should be followed.
- PATENT NOTICE. When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any invention that may in any way be related thereto.