

MIL-P-231A (NOrd)
1 September 1955
Superseding
JAN-P-231
13 April 1949

MILITARY SPECIFICATION

PROPELLANT, PYROCELLULOSE

1. SCOPE

1.1 Scope. - This specification covers smokeless propellant intended for use as a propelling charge for cannon.

1.2 Classification. - Propellant powder covered by this specification shall be furnished in the following types, as specified:

Type I - Cylindrical multiple-perforated grains
Type II - Cylindrical single-perforated grains.

2. APPLICABLE DOCUMENTS

2.1 The following specifications, standards, publications and drawings of the issue in effect on date of invitation for bids, form a part of this specification:

SPECIFICATIONS

MILITARY

JAN-D-98 - Diphenylamine
JAN-G-155 - Graphite (For Use in Ammunition)
JAN-F-193 - Potassium Sulfate (For Ordnance Use)
JAN-E-199 - Ether, Diethyl
JAN-N-244 - Nitrocellulose (For Use in Explosives)
JAN-P-270 - Powder, Propellant, Cannon
JAN-A-463 - Alcohol, Ethyl (For Ordnance Use)
MIL-L-18618 - Lead Carbonate, Basic, Dry (For Ordnance Use)

STANDARDS

MILITARY

MIL-STD-129 - Marking for Shipment and Storage

PUBLICATIONS

NAVY

OP 5 - Ammunition Ashore

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DRAWINGS

BUREAU OF ORDNANCE

Packing Box Mk 7 and Mods, except Mod 1

(Copies of specifications, standards, drawings 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 of invitation for bids, forms a part of this specification:

CODE OF FEDERAL REGULATIONS

49 CFR 71-78 - Transportation, Interstate Commerce Commission,
Explosives and Other Dangerous Articles.

(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 New Manufacture. - The raw materials used in the manufacture of the propellant shall conform to the applicable specifications listed in 2.1 for the types, grades and classes indicated below:

Diphenylamine - JAN-D-98
Graphite (For Use in Ammunition) - JAN-G-155, Grade I
Potassium Sulfate (For Ordnance Use) - JAN-P-193, Grade A
Ether, Diethyl - JAN-E-199, Grade A or C
Nitrocellulose (For Use in Explosives) - JAN-N-244, Grade A
Alcohol, Ethyl (For Ordnance Use) - JAN-A-463, Grade 2
Lead Carbonate, Basic, Dry (For Ordnance Use) - MIL-L-18618 (NOrd).

3.1.2 Rework Manufacture. - Pyrocellulose propellant may be manufactured under this specification using previously accepted pyrocellulose propellant as the base material. Prior to use, the Naval Ammunition Depot forwarding the powder furnishes evidence of satisfactory stability (60 days at 65.5 C) and this evidence is used as approval for reworking. Addition of materials described in 3.1.1 is permissible during processing to meet manufacturing and composition-al requirements.

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3.2 Composition. - Pyrocellulose propellant furnished under this specification shall be a uniform ether-alcohol colloid of nitrocellulose of standard quality. No substance, except as herein specified, shall be incorporated into the propellant or its component parts during manufacture. The propellant shall contain all of the ingredients listed below in the specified proportions:

Table I - Chemical Composition

	Web Thickness	
	0.050 inch and below	Above 0.050 inch
Nitrocellulose	100 parts	100 parts
Diphenylamine	1.0 \pm 0.10 parts	0.5 \pm 0.05 parts
Total Volatiles	*	*
Lead Carbonate, Basic	0.75 \pm 0.15 parts**	0.75 \pm 0.15 parts**

* Total Volatiles shall not exceed the maximum as shown by the curve of total volatiles on Fig. 1.

** Mandatory for Navy; optional for Army.

3.2.1 Graphite. - If a graphite coating is required, the content shall be as specified in the contract or order.

3.2.2 Potassium Sulfate. - If incorporation of potassium sulfate in the propellant is required in the contract or order, the content shall be within 0.30 parts of the amount specified.

3.2.3 Total Moisture. - The total moisture when determined by the procedure in 4.3.6 shall be 0.60 \pm 0.40 per cent.

3.3 Heat Test at 134.5°C. - When subjected to the test in 4.3.3, the propellant shall not change the color of normal methyl violet test paper to the standard salmon pink color in less than 1 hour, and shall not explode in less than 5 hours.

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3.4 Surveillance Test at 65.5°C. - Unless otherwise specified, the propellant shall pass the 65.5°C surveillance test for its particular web thickness. Acceptance, however, shall not be deferred pending completion of any surveillance test.

3.5 Ballistic Requirements. - The propellant shall comply with the ballistic requirements for the caliber of gun covered in the contract or purchase order as prescribed by the procuring agency or bureau.

3.6 Grain Requirements. - For the respective types in 1.2, the grain forms, dimensions and compressibility shall comply with Specification JAN-P-270, except for the average compressibility which shall be not less than 35 per cent. Additional restrictions on the grain dimensions may be imposed at the discretion of the contracting officer to control ignition and bulking characteristics of the propellant. The compressibility test shall only be made on special request of the Bureau of Ordnance, or at option of inspecting laboratory.

3.7 Processing

3.7.1 Process Approval. - Details of the process of manufacture and the equipment to be used by contractor shall be approved in writing by the bureau or agency concerned before the contractor proceeds with manufacture. Any deviation from the approved manufacturing process must be submitted in writing to the bureau or agency concerned and receive written approval prior to use.

3.7.2 Dehydration of Nitrocellulose. - Nitrocellulose which has been accepted for the manufacture of propellant shall be dehydrated with alcohol to thoroughly remove water, leaving no more alcohol than that required for mixing. At least 1 pound of alcohol shall be used for each pound of dry nitrocellulose in each pressing.

3.7.3 Mixing. - After dehydrating, the blocks of nitrocellulose shall be broken up and then thoroughly mixed with the solvent in suitable mechanical mixers. The necessary amount of ether to be added is determined by climatic conditions, the number and character of operations after mixing, and the caliber of the propellant that is being made; it shall be not less than 64 per cent of the total amount of solvent used in mixing. Diphenylamine of approved purity, in the amount required, shall be dissolved in ether and added to the charge. The responsibility is upon the contractor to use the proportion of solvent which with his other processes, will result in practically perfect colloid, as shown by the strand as it issues from the graining press being of uniform texture, free from spots and blemishes. The mixing shall be continued until the solvent is uniformly distributed throughout the mass. Clean scrap may be reworked separately or by addition to fresh materials in the mixers. The reworking of dirty scrap is not permissible. The necessary amount of diphenylamine, or other component required to be added to produce specified percentages in the finished propellant, shall be incorporated in each mixing charge.

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3.7.4 Pressing. - The material coming from the mixers shall be blocked and strained for the removal of lumps and any foreign material before going through, or in, the graining press. The colloid shall be pressed through dies and cut with such uniformity as will produce the standard grain required. The area of the screen holes of the die must be at least $1\frac{1}{4}$ times the area of the cross section of the die.

3.7.5 Drying. - The specification contemplates as standard practice that the propellant shall be subjected to a suitable solvent recovery process, followed by a period of drying in air-dry houses. When the invitation for bids is so worded as to allow it, proposals or alternative proposals may be submitted for the propellant dried by different processes, sufficiently described to permit their effect upon the propellant, and therefore their permissibility, to be judged. All such processes, as for instance, "water drying," must conform to the general principles of the standard process; that is, such initially mild treatment and gently progressive increase of temperature (with rigid maximum limits) as may be adjudged sufficiently favorable to the production of a first-class finished propellant. Propellant manufactured under any such allowed alternative of drying must, in the absence of special exceptions in the contract, meet all the tests herein described in order to be acceptable. The standard drying practice, required unless otherwise specified in the contract or order, shall be as follows: The propellant, after graining, may be placed at once in the solvent recovery apparatus, but the temperature and other conditions of treatment therein must be so mild at first and so gently progressive that no harmful effects shall be produced in the propellant, and in no case shall the temperature in solvent recovery exceed 55°C . The propellant shall then be finally dried in air in dry houses at a temperature not exceeding 55°C . A recording thermometer shall be suitably located in each dry house. At least two maximum and minimum thermometers shall be placed in the propellant, one in the hottest and the other in the coolest part, and daily temperature records shall be kept. Upon completion of the drying, the heat shall be turned off and the propellant allowed to cool to a temperature below 35°C .

3.7.6 Handling. - The propellant and its standard ingredients shall at all times be protected from the action of direct sunlight and acid fumes.

3.7.7 Sorting. - The propellant shall be sorted thoroughly so as to remove cracked, distorted, short and long grains and any otherwise deformed grains. This provision applies only to 8 inch grains and larger.

3.8 Description Sheets. - With every lot of propellant submitted for acceptance, the contractor shall furnish, on official blanks, eight copies of a description sheet giving a complete history of its manufacture together with chemical and physical analyses.

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4. QUALITY ASSURANCE PROVISIONS AND TEST REQUIREMENTS

4.1 Lot Size. - The lot size shall be a maximum of 165,000 pounds unless otherwise specified in the contract or order. When cross-blends of sections are requested, a maximum of 650,000 pounds will be permitted.

4.2 Sampling and Inspection. - This shall be done in accordance with Specification JAN-P-270. If the lot is made up of more than one section, the samples shall include at least one sampling from each section.

4.3 Test Procedures. - For Navy purchases, the tests shall be conducted at Government facilities unless otherwise specified in the contract or order.

4.3.1 Ballistic Tests. - Ballistic tests shall be conducted as specified by the procuring agency or bureau for the particular caliber involved.

4.3.2 Compressibility Tests. - The procedure described in Specification JAN-P-270 shall be employed to determine compliance with 3.6.

4.3.3 Heat Test at 134.5°C. - The procedure described in Specification JAN-P-270 shall be employed to determine compliance with 3.3.

4.3.4 Surveillance Test at 65.5°C. - The surveillance test shall be conducted as indicated in NAVORD O.P. 5 for Navy purchases. For Army purchases, this test is for information purposes only.

4.3.5 Form, Dimensions and Compressibility of Grains. - Compliance with 3.6, when required, shall be determined using the procedures described in Specification JAN-P-270.

4.3.6 Total Moisture. - Transfer a weighed portion of 100 to 200 gm. of the sample to a 500-ml. balloon flask and add 200 ml. of carbon tetrachloride. Fill the graduated portion of a moisture tube (see fig. 2) with carbon tetrachloride and attach the tube to the flask and condenser of the Allihn type. Standard taper ground-glass joints should be used whenever possible in apparatus shown in figure 2. Connect a calcium chloride tube to the top of the condenser to keep out atmospheric moisture. Heat the flask by means of a suitable steam or hot water bath so that the distillate falls from the end of the condenser at the rate of two to three drops per second. Continue the distillation until the water layer in the measuring tube remains constant for three successive readings taken at 30-minute intervals. Disconnect the apparatus and wash the water layer with fresh carbon tetrachloride from a burette at the rate of approximately 5 ml. per minute, until the volume of the water layer becomes constant. This usually requires 25 to 50 ml. of carbon tetrachloride. Read the point of contact of the top meniscus with the wall of the tube and the highest point of the

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lower meniscus. Determine the difference in these readings and record this as the volume of water found.

$$\text{Percent total moisture} = \frac{100 V}{W}$$

where

V = volume of water
W = weight of sample.

In the event that the volume of water distilled over is 0.2 ml. or less, a satisfactory reading cannot be obtained. In this case, add, by means of an accurate pipette, 1.00 ml. of water to the graduated portion of the measuring tube, then read, and correct for the volume of water added.

4.3.7 Chemical Analyses. - Since the chemical composition of the propellant will vary with the addition or omission of the optional materials potassium sulfate, lead carbonate and graphite, the requirements in 3.2 have been shown as parts instead of per cent. The analytical procedures described in subsequent sections, however, yield results as percentages. For purposes of determining conformance with the requirements of 3.2, 3.2.1 and 3.2.2, corrected values calculated in accordance with 4.3.7.6 shall be used.

4.3.7.1 Diphenylamine - New Powder. - Weigh accurately a sample of 5 gm. of the propellant into a 250-ml. lipped beaker. The sample may be in whole grains, if small enough to give a fair sample, or in slices of medium or larger grains. Mix 10 ml. of glacial acetic acid and 20 ml. of nitric acid (sp. gr. 1.42) and pour on the propellant. Cover the beaker with a watch-glass and place on a steam bath at approximately 95°C for 1½ hours. It is necessary that the above quantities of acid be strictly adhered to, by using either a pipette or graduate for the measurement. The powder dissolves completely with copious evolution of red fumes. The time and temperature given will allow all red fumes to be dissipated without too much reduction of volume, and this is the end to be attained, as the nitro compounds have a tendency to crystallize out if the solution is evaporated too much or it is allowed to stand too long after removal from the steam bath. Whenever such crystallization occurs, results are slightly low. Immediately after heating on the steam bath, cool the solution, being careful not to agitate, and pour into 75 ml. of distilled water which has been cooled to 15°C in a 250-ml. glass-stoppered Erlenmeyer flask. Wash the beaker with water so that the solution, including washings, will be approximately 120 ml. Shake the flask well for about 2 minutes and allow to stand overnight. Filter the nitro compound on an asbestos mat type filtering crucible prepared by washing with 10-per cent nitric acid and igniting. Wash the nitro compound six or seven times with water containing 1 per cent of nitric acid, dry at 100° to 105°C for 1 hour, cool in a desiccator, and weigh. Place the crucible in a small beaker, add 10 ml. of acetone, and allow to soak for 15 minutes. Place the crucible on a suction flask and wash with small quantities of acetone until nitro compounds are completely removed. Dry the crucible at 100° to 105°C for 1 hour, cool in

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a desiccator, and weigh. Calculate the loss in weight as nitro compounds. The factor for conversion of nitro compound to diphenylamine, using the quantities of acid stated above, is 0.4259.

$$\text{Percent diphenylamine} = \frac{42.59 A}{W}$$

where

A = weight of nitrocompound

W = weight of propellant.

4.3.7.2 Diphenylamine - Reworked Powder. - Transfer 5 gms. of the ground sample (20 mesh) to a 1 litre Ladenburg flask and add 200 ml. of 15% NaOH solution. Connect flask to the condenser which projects into a 1 litre separatory funnel. Pass a current of steam through the NaOH solution and distill at a rate of approximately 7 ml. per minute, see figure No. 3. Maintain the volume in the sample flask at approximately 200 ml. The temperature of the distillate should not exceed 30°C. After approximately 800 ml. of distillate have been collected in a 1000 ml. separatory funnel (time will require about 90 min.) disconnect the condenser and steam generator. Add 75 ml. of methylene chloride to the distillate through the condenser. Remove the condenser. Add 10 gms. of NaCl to the distillate. Stopper the funnel and shake vigorously for one minute. Allow the liquids to separate for approximately 15 minutes and transfer the methylene chloride layer to a 500 ml. iodine flask. Repeat the extraction two additional times with 50 ml. portions of methylene chloride. Collect the solvent fractions in the 500 ml. iodine flask. Evaporate the solvent in a well-ventilated hood with a stream of dry air. Dissolve the residue in 75 ml. of 70% acetic acid and cool to 15°C. Add, by pipette, 25 ml. of 0.2 N bromide-bromate solution. Add 10 ml. of 1:1 HCl and stopper the flask immediately. Seal the stopper with a few drops of 15% KI solution and swirl the flask and contents for 75 to 90 seconds. Immediately add 15 ml. of 15% KI solution through the funnel neck of the flask. Avoid venting the flask during the addition of the KI. Swirl the flask and contents with the stopper in place, and then allow to stand for 2 minutes and titrate with 0.1 N standard $\text{Na}_2\text{S}_2\text{O}_3$ to a starch end point. Make a blank determination on 75 ml. of 70% acetic acid employing the same procedures and reagents used for the sample determinations.

$$\text{Percent available diphenylamine} = \frac{2.115 N(B-V)}{W}$$

where

B = ml. $\text{Na}_2\text{S}_2\text{O}_3$ consumed by blank titer

V = ml. $\text{Na}_2\text{S}_2\text{O}_3$ consumed by sample titer

N = normality of $\text{Na}_2\text{S}_2\text{O}_3$

W = weight of sample

4.3.7.2.1 Solutions Required. -

(a) 15% KI solution - Dissolve 15 grams of KI crystals in 100 ml. of distilled water. Prepare fresh daily.

(b) 70%, by volume, acetic acid - Mix 70 ml. of glacial acetic acid with 30 ml. of distilled water.

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4.3.7.4.1.2 Preparation of solution tubes. - Place 10 clean dry steel balls, 5/16 inch in diameter, and 50 ml. of dibutylphthalate reagent into each of a series of clean dry solution tubes (see fig. 3). The dibutylphthalate reagent may be added through a small funnel either by means of a graduated cylinder or from an automatic 50-ml. delivery device provided with a larger-normal outlet. Clean the inlet of each solution tube with absorbent cotton held by steel forceps. Using the forceps, insert a wad of pyrex glass wool, or equivalent, (about 0.2 gm.) in the inlet of each tube at such a point that it will not be in danger of falling into the tube and at the same time will not be in too close contact with the cork stopper that is to be subsequently inserted. By means of 5-cm. pieces of rubber tubing¹ attach each solution tube to the manifold of a rocking device inside an oven² maintained at 85°C. Attach to the manifold a vacuum line assembly³ (see Fig. 4) and a vacuum pump which is capable of consistently maintaining a pressure

¹Rubber pressure tubing should have a 5/16-inch bore, 3/16-inch wall. Use 5-cm pieces for connecting the glass tubes to the copper manifold. Cut off a number of pieces, boil these in 5-percent sodium hydroxide solution for 10 to 15 minutes, wash thoroughly, and dry at 100°C. The rubber tubing should be examined after each total volatile determination and replaced when necessary.

²The oven should be fitted with a rocking device and electric motor. By means of the rocking device the tubes are tilted back and forth through an angle of approximately 90° at the rate of 12 complete cycles per minute. The oven designated as Catalog No. 95105-A by the Central Scientific Co. has been found satisfactory and should be provided with a safety latch to replace the regular latch. A steam oven, if available, may be used in place of an electric oven.

³Vacuum line assembly should be as shown on figure 4. The air and vapors from the solution tubes are passed through a T-tube leading to a McLeod gage, a T-tubing leading through an aspirator bottle I, filled with drierite, to a needle valve E, a three-way stopcock F, a 500-ml. absorption bottle H containing silica gel, a trap D, a T-tube leading to a stopcock G, and a pressure regulator C. The bottle of drierite I serves to dry the air which is admitted to the tubes in the oven. The needle valve E permits a much closer control of the pressure during the initial stage of the evacuation than can be obtained by stopcock E. The three-way stopcock F serves to admit nitrogen to the tubes in the oven. The absorption bottle of silica gel H is included to absorb the volatiles from the powder and thus make it unnecessary to change the oil in the vacuum pump frequently. The silica gel may become ineffective as shown by the inability of the vacuum pump to exhaust the system to 1 mm. or less. If this happens, the silica gel should be replaced with fresh material. The trap D serves to keep dibutylphthalate reagent, in the pressure regulator, away from absorption bottle H in case stopcock A is accidentally closed when the vacuum pump is stopped. The stopcock G serves to admit air into the vacuum pump and pressure regulator after the tubes have been filled with nitrogen. The regulator G, containing dibutylphthalate reagent serves to maintain the desired interior pressure.

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- (c) Starch indicator - Slurry 2 grams of soluble starch in 100 ml. of cold distilled water. Pour the slurry into 1900 ml. of boiling water. Cool. Add 0.1 gm. of mercuric iodine to stabilize the solution.
- (d) 1:1 HCl - Dilute 50 ml. of reagent grade concentrated HCl with 50 ml. of water.
- (e) 0.2 N bromide bromate solution - Dissolve 30 grams of KBr, 5.5 grams of KBrO_3 , and 1 gram of KOH in 1 liter of distilled water.
- (f) 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution - Dissolve 25 grams of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ and 0.2 gms. of Na_2CO_3 in one liter of water. Allow the solution to stand 1 week before standardization.

4.3.7.2.2 Standardization. - Weigh accurately 0.2 grams of NBS $\text{K}_2\text{Cr}_2\text{O}_7$ and transfer it to a 250 ml. iodine flask. Dissolve the crystals in 50 ml. of distilled water. Add 10 ml. of 1:1 HCl and 15 ml. of 15% KI solution; stopper the flask and allow to stand for 5 minutes. Titrate with the $\text{Na}_2\text{S}_2\text{O}_3$ solution to a starch end point.

$$\text{Normality of } \text{Na}_2\text{S}_2\text{O}_3 = \frac{W}{.049035 V}$$

where

W = weight of $\text{K}_2\text{Cr}_2\text{O}_7$
 V = ml. of $\text{Na}_2\text{S}_2\text{O}_3$ consumed

4.3.7.2.3 Apparatus Required. -

- (a) Ladenburg distilling flask, Capacity 1000 ml.
 (b) Separatory Funnel, capacity 1000 ml.
 (c) Spiral Water Condenser, Electric Heater, Central Scientific Co. No. 16530, 250 watts.
 (d) Electric Heater, Central Scientific Co. - No. 16595, 750 watts.

4.3.7.3 Graphite. - Place the crucible used for the determination of diphenylamine (see par. 4.3.7.1) in a muffle furnace or in an inclined position over a gas burner and heat strongly until all carbonaceous material has disappeared. Cool the crucible in a desiccator and weigh. Calculate the loss in weight as percentage of graphite in the sample as received.

4.3.7.4 Total Volatiles. -

4.3.7.4.1 Preparation for test. -

4.3.7.4.1.1 Dibutylphthalate reagent. - Prepare a large quantity of dibutylphthalate reagent containing approximately 1 gm. of diphenylamine per liter as follows: Dissolve the desired quantity of diphenylamine in a small quantity of hot dibutylphthalate. Pour this solution into a large volume of dibutylphthalate and shake this solution vigorously. When tested separately in the solution-evacuation apparatus it should lose less than 10 mg. It may be necessary to pretreat the dibutylphthalate as follows to meet this requirement. Heat the dibutylphthalate for 2 hours at 145° to 150°C while bubbling dry air through it.

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of 1 mm. or less. Close stopcock C. With stopcock A open and stopcock B and needle valve E closed, turn on the rocking motor and vacuum pump, and evacuate the tubes to a pressure of 1 mm. or less as indicated by the McLeod gage. This pretreatment procedure is accomplished very easily because the evacuation is started when the dibutylphthalate reagent is still at approximately room temperature. The low boiling impurities are removed gradually at 1 mm. pressure as the temperature rises. At the end of 1½ hours, stop the vacuum pump and rocking motor, open stopcock B cautiously, and admit dry air slowly by gradually opening the needle valve E. If air is admitted too rapidly, the wads of pyrex wool, or equivalent, may be drawn into the solution tubes. Wearing a pair of gloves, remove the solution tubes from the oven, leaving the 5-cm. rubber connecting tubes attached, and place the solution tubes in a wire-screen tray (see Fig. 5). Immediately insert small protective drying tubes¹ (see Fig. 6) and allow the tubes and contents to cool to room temperature. This will require from 30 to 45 minutes. The small drying tubes will prevent the admission of atmospheric moisture during the cooling period.

4.3.7.4.1.3 Determination of loss of weight of solution tubes. - The determination of the loss in weight of a solution tube involves the measurement of a decrease of 20 to 40 mg. (for powders containing 1 to 2 percent total volatiles) in the weight of a 170 gm. system. Therefore, the weighing and conditioning procedure is very important. After the tubes have cooled to room temperature, keep them stoppered, except while being weighed, to prevent absorption of atmospheric moisture by the very dry dibutylphthalate reagent.

Remove the rubber connectors and protective drying tubes from all of the solution tubes and insert No. 1 cork stoppers. Wipe the tubes with a wet towel and then wipe (but do not rub) with a clean dry cloth which is substantially free from lint. This operation is for the purpose of cleaning the tubes, removing electrostatic charges, and hastening the attainment of equilibrium with moisture content of the air. Place the solution tubes in a wire-screen tray, cover them with a cloth, and allow them to stand near the balance for at least 30 minutes. At no time after a tube has been conditioned should it be touched by the fingers.

¹Small protective drying tubes—16 are required. These tubes contain indicating drierite (or equivalent). Store them in a desiccator when not in use. Keep the drierite anhydrous (blue) by occasionally putting the tubes in an oven at 130° to 150° overnight.

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In putting a tube on the balance to weigh it, handle it only by its inlet tube after covering the latter with a small piece of tissue paper. Place each solution tube in a wire tube holder (see fig. 7) and weigh.

4.3.7.4.2 Test Procedure

4.3.7.4.2.1 Small Grains. - For specimens consisting of small grains where each grain weighs less than 0.2 gm. take approximately 2 gm. of whole grains of propellant, for each determination. weigh the samples to the nearest 0.01 gm. in an aluminum weighing dish, place them in a series of small weighing bottles, and stopper the bottles immediately. weigh 2 or 3 of the tubes accurately for use in making blank determinations; i.e., determination of the small change in weight that occurs when the tubes of dibutylphthalate reagent are heated and evacuated under the same conditions that prevail in the analysis, make these weighings, as well as all other accurate ones, by using a counterpoise¹ (fig. 8) on the righthand side of the balance. Keep this counterpoise near the balance so that it will be practically in equilibrium with the prevailing conditions of temperature, pressure, and humidity. Do not wipe the counterpoise when the solution tubes are wiped. Make at least two blank determinations (the first and eighth tubes) if a total of 8 tubes are being used, but make at least three blank determinations if a total of 16 tubes are being used (the first, eighth, and sixteenth tubes). Add the weighed samples to the tubes and weigh the tubes accurately. From each tube remove the cork stopper, remove the wad of pyrex wool with forceps, add the sample with the aid of a small metal funnel having an outlet of approximately 9 mm. outside diameter, reinsert the wad of pyrex wool, or equivalent, weigh the tube accurately, shake it if necessary to loosen any grains which may be adhering to the walls of the tube, connect it to the manifold in the electric oven, and rock the tube mechanically. It is considered desirable, although not essential, to have the samples dissolve completely during the overnight rocking period at 85°C and they are much more likely to dissolve if the steel balls and grains of propellant all move freely in the tubes when they are put in the oven. If the samples are of such a nature that they do not adhere firmly to the walls of the tubes on standing in the dibutylphthalate solution for 30 minutes (this must be determined by experience), the procedure just described may be shortened in two respects:

1

A counterpoise tube is a glass tube containing a suitable number of steel balls so that it will have approximately the same weight, volume, and exterior surface as a solution tube containing 50 ml. of dibutylphthalate. This tube should be kept hanging near the balance, covered with a cloth to protect it from dust.

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(1) the weighed samples may be transferred directly from the aluminum weighing dish to the solution tubes and (2) the whole series of tubes may be put into the oven at the same time. In some cases it has been found advantageous to rock the weighed solution tubes in a cradle from the time they are weighed so that all can be put into the oven at the same time.

4.3.7.4.2.2 Large grains. - For specimens consisting of large grains, where each grain weighs 0.2 to 0.4 gm., take one-half of each of 20 to 10 grains, so that each sample will weigh approximately 2 gm., for each determination. These small grains may be cut with heavy tin snips inside a 1-gallon container if desired, but it is preferable to cut several grains at a time with a powder cutter¹ (fig. 9). If each grain weighs more than 0.4 gm., cut approximately 4 gm. from the centers of 10 grains with the powder cutter. Immediately after cutting the slices, place them in a weighing bottle and stopper it at once. When the slicing operation has been completed, subdivide the slices with tin snips so the fragments can be easily placed in solution tubes. Divide the fragments into 2 approximately equal portions and put each one into a stoppered weighing bottle. Then transfer the contents of the weighing bottles to the solution tubes which have previously been weighted to the nearest 0.01 gm. All of this work should be done with forceps so that touching the powder grains with the fingers is avoided. It should be completed as quickly as possible to avoid excessive loss of total volatiles. As soon as all the tubes have been placed in the oven, turn on the rocking motor. Open the nitrogen cylinder valve to allow a slow stream of nitrogen to escape through the mercury trap. With stopcock A open and stopcock B closed, evacuate the tubes, fill them with nitrogen, evacuate them a second time, and again fill them with nitrogen. This is accomplished very easily by manipulating stopcock F. The nitrogen should be admitted rather slowly to prevent the gas stream carrying any particles of foreign matter from the rubber tubing or manifold into the tubes. Leave stopcock F turned to connect the oven with the tube leading to the nitrogen overflow bottle containing the mercury. Then admit air to the pressure regulator, vacuum pump, etc., by opening stopcock G. The operation of filling the tubes with nitrogen is facilitated by having them at approximately room temperature at this point so that they can be evacuated quickly without taking any special precautions. Rock the tubes at a temperature of 85°C. for approximately 15 hours. Ordinarily the samples dissolve in the dibutylphthalate reagent under these conditions. It is essential that the samples be softened by dibutylphthalate reagent, but not essential that they dissolve completely. After the tubes have been rocked for approximately 15 hours,

¹A powder cutter is a modified paper cutter with a 16-inch blade. The stationary guide plate adjacent to the blade has been provided with 16 holes ranging in size from one-eighth to three-fourth inch in which to insert the propellant grains. A movable guide plate behind the stationary plate but parallel to it is actuated by a screw. The movable plate is provided with pins which fit into the holes in the stationary plate when the two plates are brought together. To aid in cutting thin uniform slices, the wheel actuating the movable plate is graduated on its circumference in small divisions. By rotating the wheel one small division to the left, the movable plate is moved forward 0.17 mm. By inserting grains in the holes of the stationary plate, turning the wheel the proper amount, and operating the cutting lever, slices of any desired thickness can be cut.

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remove the volatile matter by evacuation at 5 mm. pressure for 2 hours. The beginning of the evacuation, while most of the volatile matter is being removed, is the critical point of the determination. Assuming that the tubes are properly conditioned and weighed, the accuracy of the results depends upon the care with which this operation is performed. With the rocking device in operation and with stopcocks A and B and the needle valve E open, turn on the vacuum pump. Evacuate at gradually decreasing pressures by slowly closing needle valve E until a pressure of 5 mm. is reached. The lowering of the pressure from about 50 mm. to 5 mm. must be done cautiously over a period of approximately 10 minutes while watching the operation through the glass door of the oven. Close stopcocks A and B, and continue the evacuation for 2 hours at 85°C and 5 mm. The pressure must be reduced so gradually that bubbles of volatile matter leave the dibutylphthalate solution without danger of causing mechanical loss. At the end of 2 hours, stop the vacuum pump and rocking motor.

Open stopcocks A and B, and gradually admit air into the system through needle valve E. If stopcock A is not opened at this point, the dibutylphthalate solution will flow out of the pressure regulator. Remove the tubes from the oven, insert the protective drying tubes in the rubber connectors, let the tubes cool to room temperature and insert cork stoppers. Condition and weigh the tubes in the same manner as that previously described. If a special ink has been used instead of hydrofluoric acid solution to put numbers on the tubes, care must be taken to avoid rubbing off any of the ink. As a matter of good practice and in order to obtain minimum blank values, the conditioning procedures before and after the removal of the volatile matter should be similar.

$$\text{Percentage of volatile solvents} = \frac{100 (A+B)}{W}$$

where

A = decrease in weight of sample tube

B = average change in weight of blank tubes

W = weight of sample

The changes in weight of the blank tubes are usually negative, but may be positive (possibly due to humidity changes): They seldom exceed 3 mg. In all cases the average change in weight, B, is algebraically added to A; i.e., if B is negative it is subtracted from A, if positive it is added. Blank values are usually concordant within 1 mg. If they are not concordant within 2 mg.; the analysis should be repeated. This method has a precision of 0.10 percent, i.e., results should agree within 0.01 unit when expressed as percent total volatiles. If they do not agree this closely, the analysis should be repeated.

4.3.7.4.3 Alternate total volatiles test. - Start the volatile determination as soon after receipt of the sample as possible. Do not return to the sample container any grains which have been handled, except in special cases where the sample available is very small. Keep the sample container tightly closed. Use about 20 grains of all propellants, regardless of caliber. Cut a total of at

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least 2 gm., taking approximately the same number of slices from each grain, after first cutting off and discarding approximately one-fourth of the grain, so that the slices used come from approximately midway between the end and center of the grain. Cut each slice so as to have, as nearly as possible, the same thickness at all parts of its area. Cut the slices either clear across the grain giving a circular area, or just half across, giving a semicircular area. Weigh accurately, as quickly as possible, two 1 gm. samples prepared as described above. Place these samples in 400-ml. beakers, cover each with 150 ml. of an ether-alcohol solution (2 ether-1 alcohol) prepared from solvents redistilled before being mixed. Cover each beaker with a watch glass and allow to stand overnight under a bell jar resting on a ground glass plate. In the morning agitate the solution until the propellant is dissolved. Place the beaker on a steam bath, boil off part of the solvent, and add approximately 50 ml. of water to precipitate the nitrocellulose, the solution being stirred vigorously after the first addition of water until precipitation is complete. The exact quantity of water to effect the precipitation and the method of adding it are governed by the behavior of the precipitate and by the personal preference of the operator, but the total quantity of water added must be 50 ml. In general, the more solvent boiling off and the larger the quantity of water added the coarser the precipitate. Some analysts prefer to add part of the water before boiling the solution. The most desirable form of precipitate consists of flakes approximately one-sixteenth inch in diameter, or strings one-eighth to one-fourth inch long. After the precipitate has been thrown down, evaporate the liquid on the steam bath. Weigh together a 2½-inch diameter glass evaporating dish and a 2 by 1-1/8-inch weighing bottle, transfer the dry precipitate, as far as possible, to the bottle and wash that which adheres to the beaker into the dish, using 50 ml. of water and scrubbing the beaker thoroughly with a rubber policeman. Place the dish and bottle together at 100°C. in an oven for 1 hour, cool in a desiccator, and weigh. Heat again for one-half hour, cool, and reweigh. If the latter weighing does not check the former within 0.0005 gm. repeat the one-half hour dryings in the oven until the results do check within this limit. Run a nonvolatile residue at 95° to 100°C on the quantities of water, alcohol, and ether used and calculate the percentage of total volatiles as follows:

$$\text{Percentage of total volatiles} = \frac{(A+B-C) 100}{W}$$

where

- A = loss in weight of the sample
- B = weight of water, ether, and alcohol residues
- C = correction in grams for volatilization of diphenylamine during the determination. Allow 25 percent of the diphenylamine content of the powder
- W = weight of sample

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4.3.7.5 Basic Lead Carbonate. - Transfer a 10 gram sample of the propellant, previously ground in a Wiley mill to pass a 20 mesh screen, into a 250 ml. beaker and add 50 ml. of concentrated nitric acid. Cover with a watch glass and place on the steam bath. After decomposition of the sample is completed (approximately $\frac{1}{2}$ hour is sufficient for all propellants containing lead compounds tried), add 20 ml. of 1:1 sulfuric acid and put a rubber ring around the beaker so that it can be held at a level about half way down into the steam bath; cover the beaker with a ribbed watch glass and leave it for one hour - this length of time has been found sufficient to remove most of the nitric acid with no charring in the solution. Remove from the steam bath and while the solution is still hot, add isopropyl alcohol dropwise until the initial vigorous reaction subsides. Then add a few ml. of the isopropyl alcohol in excess. (Isopropyl alcohol does not form dangerous nitration products. It also reduces the residual nitric acid more efficiently in a sulfuric acid medium of 60% or higher. The amount of isopropyl alcohol varies considerably with different types of propellants.) Add 100 ml. of 50% ethanol, heat on a steam bath for about 10 minutes and filter on a fine porcelain crucible. Wash with acetone until the filtrate comes through colorless and then wash with 50% ethanol. Ignite at 500 to 600°C for $\frac{1}{2}$ hour. Powders containing graphite must be filtered after the nitric acid digestion due to reduction of lead sulfate at ignition temperature.

4.3.7.5.1 Modification for propellants containing graphite. - After the nitric acid digestion, add 50 ml. of water and leave on the steam bath until the graphite has coagulated. At this point sufficient nitric acid should be present in the mixture to hold in solution any lead sulfate forming in propellants which originally contained potassium sulfate. In some cases it may be necessary to add more nitric acid. Filter the hot solution rapidly through a Whatman No. 1 paper into a 250 ml. beaker and wash with hot 1% nitric acid. Add 10 ml. concentrated sulfuric acid to the filtrate and continue as above, except that approximately 2 hours in the steam bath will be required to remove sufficient water and nitric acid so that the last traces of nitric acid can be removed by reduction with isopropyl alcohol.

Calculations:

$$\text{Percent basic lead carbonate} = \frac{\text{Wt. of PbSO}_4 \times 100 \times 0.8526}{\text{Wt. of sample}}$$

4.3.7.6 Potassium Sulfate. - Transfer a weighed sample of approximately 10 gm. of ground or sliced powder to a 400 ml. beaker. Add 20 ml. of concentrate nitric acid, cover and heat on the steam bath. After the initial reaction has subsided, remove the cover and evaporate almost to dryness. Add 10 ml. of 38% hydrochloric acid and evaporate almost to dryness. Repeat the evaporation with hydrochloric acid. Add 100 ml. of water and heat on the steam bath. Add 15 ml. of 10% barium chloride slowly with stirring. Digest the precipitate one hour, then filter hot through No. 42 Whatman paper. Wash the precipitate thoroughly with hot water. Place the filter paper and precipitate in a freshly-ignited and tared crucible and dry in the oven. Then ignite at 800-900°C. Cool and weigh.

$$\text{Percent potassium sulfate} = \frac{\text{Weight of precipitate} \times 74.65}{\text{Wt. of sample}}$$

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4.3.7.7 Correction Factor. - In order to correct the percentage values to parts the following correction factor shall be used:

$$\text{Correction Factor} = \frac{100}{100-A-B-C-D-E}$$

where

- A = percent Diphenylamine from 4.3.7.1
- B = percent Graphite from 4.3.7.3
- C = percent Total Volatiles from 4.3.7.4
- D = percent Basic Lead Carbonate from 4.3.7.5
- E = percent Potassium Sulfate from 4.3.7.6.

The percent values from 4.3.7.1, 4.3.7.3, 4.3.7.4, 4.3.7.5 and 4.3.7.6 shall be multiplied by this correction factor and the resulting values checked for compliance with the requirements specified in 3.2, 3.2.1 and 3.2.2.

4.4 Resubmitted Lots. - Should a lot fail to pass the specified tests (either ballistic, chemical or stability), it shall be rejected but it may, upon application from the manufacturer and approval by the bureau or agency concerned, be resubmitted once (see par. 6.5).

4.5 Non-Valid Tests. - Should a lot fail to meet the specified test requirements (either ballistic, chemical or stability), a second test shall be authorized only if the failures can be ascribed to deficiencies in the testing conditions. Data and information substantiating the requested second test shall be submitted to the procuring agency for review. If a second test is authorized by the procuring agency, the first test shall be discounted and the results of the second test shall be used to determine lot disposition.

5. PREPARATION FOR DELIVERY

5.1 Packing and Packaging. - The requirements of JAN-P-270 shall be followed for this material.

5.2 Marking. - In addition to any special marking required by the contract or order, shipments shall be marked in accordance with specification JAN-P-270, standard MIL-STD-129 and the Interstate Commerce Regulations for Transportation of Explosives (2.2).

6. NOTES

6.1 Use. - The propellant covered by this specification is intended for use as a propelling charge for guns.

6.2 Procurement documents should specify the following:

- Title, Number and Date of the specification
- Type of propellant grain required (see par. 12).

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When graphite coating is required, the graphite content required (see par. 3.2.1)

When potassium sulfate addition is required, the potassium sulfate content required (see par. 3.2.2)

Average grain dimensions of Type I and II propellants (see par. 3.6)

Ballistic requirements applicable to the propellant (see par. 3.5)

Place of inspection (see pars. 4.2 and 4.3)

Process approval (see par. 3.7.1)

Information concerning government material if such is furnished.

6.3 In case the propellant fails to pass the ballistic tests in the weapon for which it is intended, the bureau or agency concerned may, if deemed to its interest, accept the propellant, upon its meeting the specifications of the bureau or agency concerned for any other weapon.

6.4 The government reserves the right to make such test, by side impact or otherwise, of the physical structure of grains, perfection of colloid, etc., as experience may dictate. Should a propellant fail in minor respects, excellence of physical structure, perfection of colloid, etc., will be considered circumstances in favor of the propellant, and imperfection in these respects will be considered as adverse.

6.5 Payment for Resubmitted Lot Tests. - All expenses incident to the testing of resubmitted lots should be borne by the contractor, even if finally accepted for the weapon for which originally intended. Ballistic, chemical, or stability samples representing finally rejected lots should not be paid for under any condition. Surveillance samples requested individually or as part of the chemical and stability sample should be paid for only if the lot is finally accepted.

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 patented invention that may be in any way related thereto.

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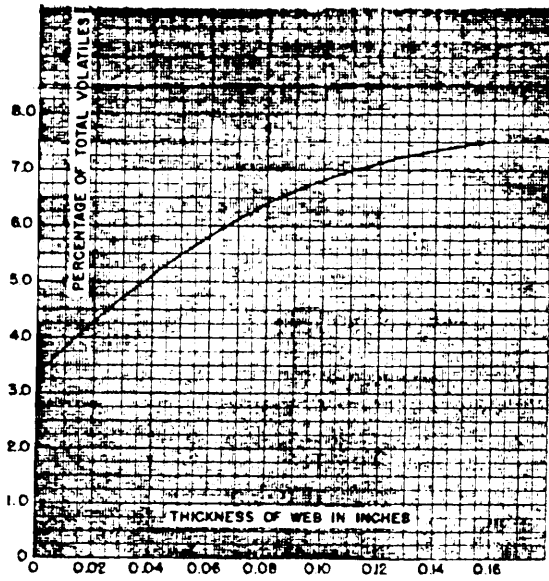


FIG. 1 TOTAL VOLATILE CONTENTS OF PYROCELLULOSE POWDERS

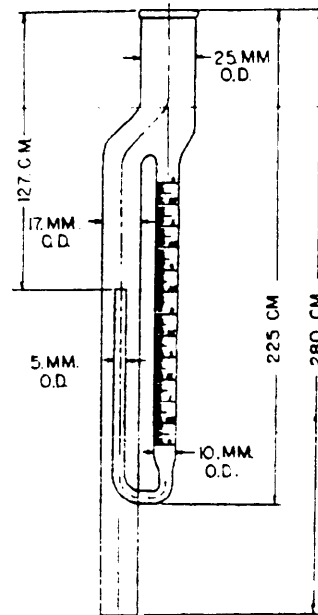


FIGURE 2.—Moisture tube.

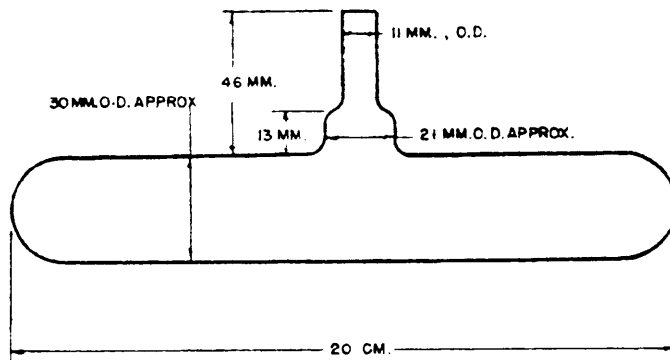
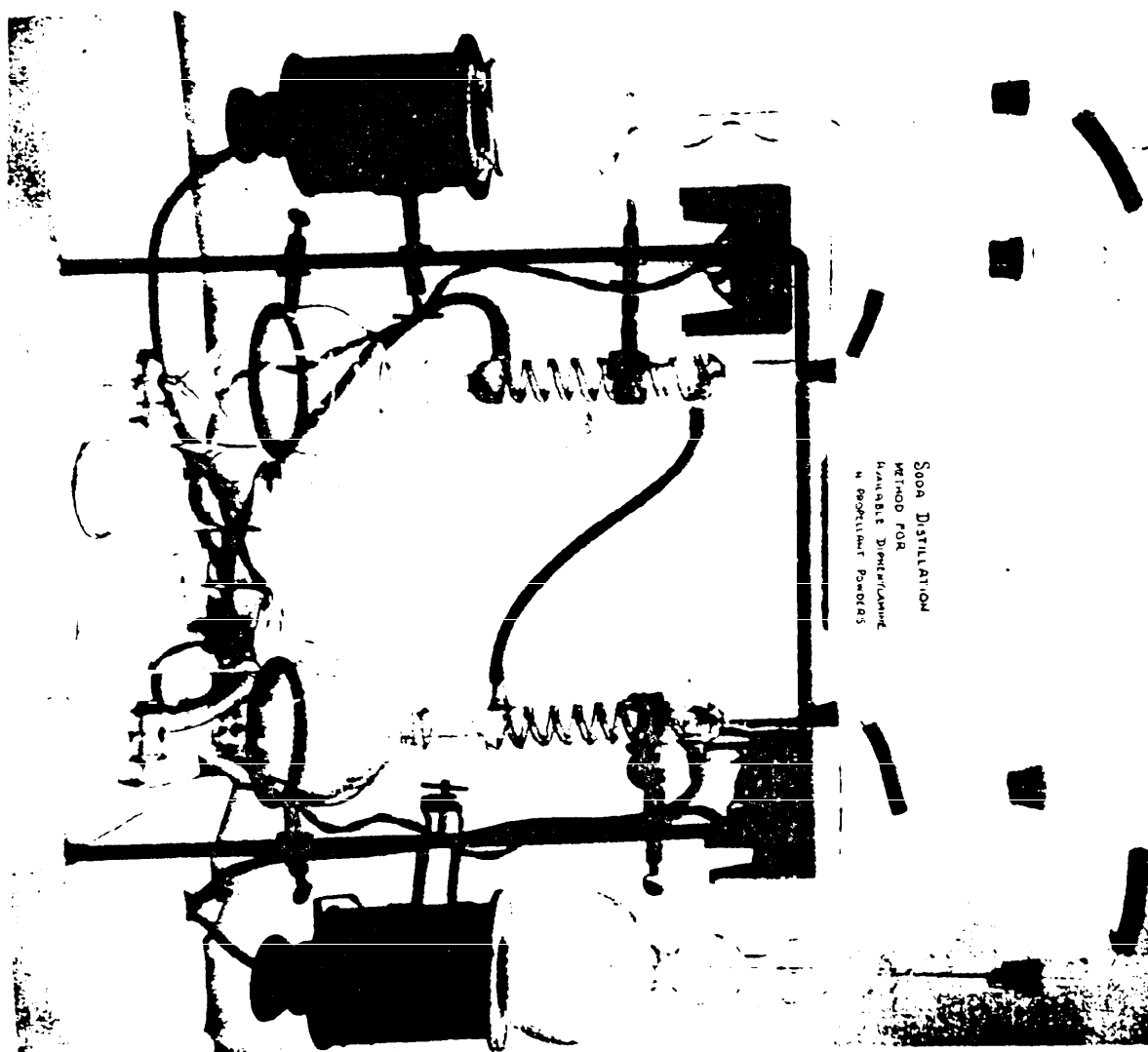


FIGURE 4—Solution tube.

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*Figure 3.
Distillation Apparatus*

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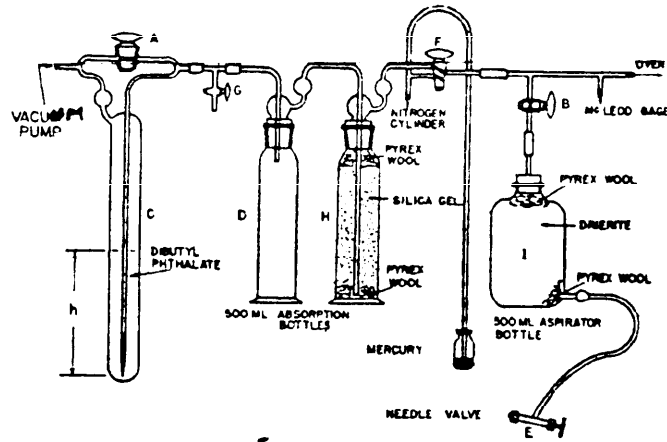


FIGURE 5—Vacuum line assembly.

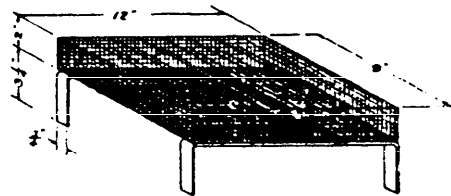


FIGURE 6—Wire screen tray.

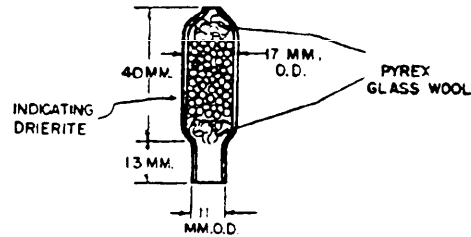


FIGURE 7—Small protective drying tube.

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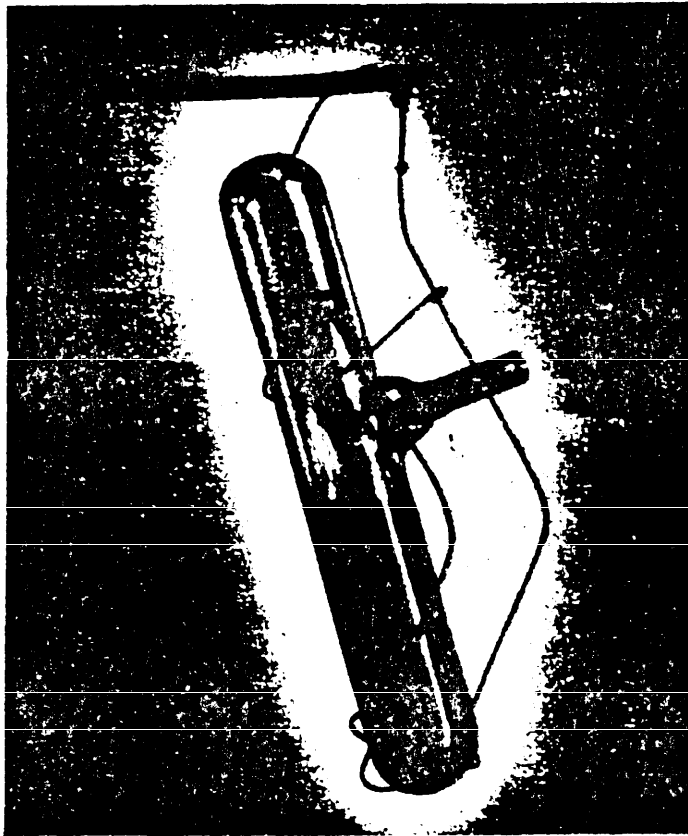


FIGURE 2—Wire tube holder.

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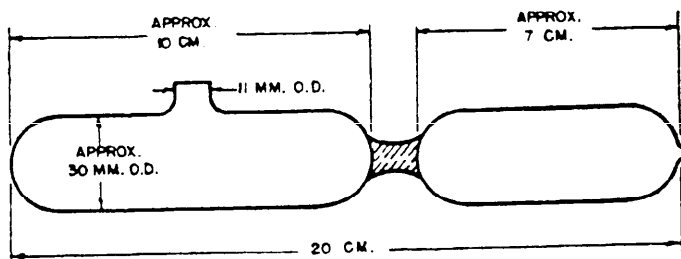


FIGURE 9—Counterpoise.

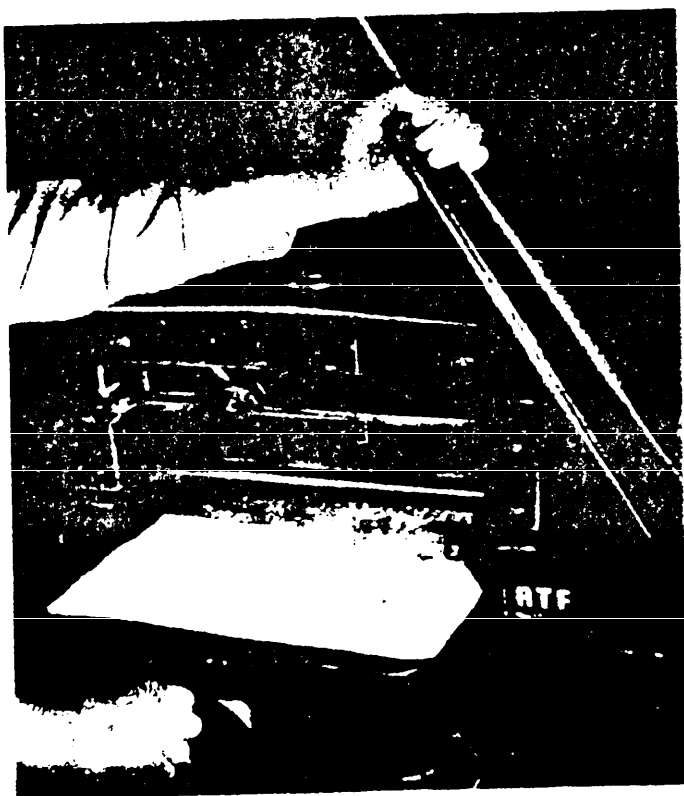


FIGURE 10—Smokable powder cutter.

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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 (as indicated on reverse herof).

SPECIFICATION

ORGANIZATION (of submitter)

CITY AND STATE

CONTRACT NO.

QUANTITY OF ITEMS PROCURED

DOLLAR AMOUNT

\$

MATERIAL PROCURED UNDER A

DIRECT GOVERNMENT CONTRACT

SUBCONTRACT

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.

COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID

IS THE SPECIFICATION RESTRICTIVE?

YES

NO IF "YES", IN WHAT WAY?

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