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MILITARY SPECIFICATION

LEAD AZIDE RD-1333

(For use in ammunition)

This specification covers a grade of lead azide to be used in detonators and/or priming composition.

1. SCOPE

1.1 This specification covers lead azide RD-1333 for use in the loading of detonators and fuzes, and in the manufacture of priming compositions.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

MILITARY

MIL-S-51132 - Sodium Carboxymethyl Cellulose (For Ammunition Use)

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes (ABC-STD-105)
MIL-STD-129 - Marking for Shipment and Storage
MIL-STD-650 - Explosive: Sampling, Inspection and Testing

FSC: 1376

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MIL-STD-1235 - Single and Multilevel Continuous
Sampling Procedure and Tables for
Inspection by Attributes.

DRAWINGS

ARMY

73-2-335 - Detonator M47 Loading Assembly and Details.

(Copies of specifications, standards, drawings and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the Contracting Officer.)

2.2 Other publications.-The following document forms a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

CODE OF FEDERAL REGULATIONS

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

(The Interstate Commerce Commission Regulations are now a part of the Code of Federal Regulations (1949 Edition and Revisions) available from the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402. orders for the above publication should cite "49 CFR 71-90 (latest revision) ").

3. REQUIREMENTS

3.1 Crystal growth control agent.-Lead Azide RD-1333 shall be precipitated from a solution of Sodium Carboxymethyl cellulose (NaCMC) which shall be used as the crystal growth control agent. The NaCMC shall be in accordance with Military Specification MIL-S-51132.

3.2 Physical and chemical characteristics.-The lead azide RD-1333 shall have the following chemical and physical characteristics when tested as specified herein.

3.2.1 Color.-The color shall be white to buff, when tested as specified in 4.3.1.

3.2.2 Form

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3.2.2.1 Aggregates.-The aggregates shall contain no well-defined translucent crystals when examined microscopically (see 4.3.2) similar to Figure 6.

3.2.2.2 Particles.-The particles shall be opaque, free-flowing powder and irregular in size and shape when tested as specified in 4.3.2.

3.3 Chemical tests

3.3.1 Purity.-The purity shall be 98.5 percent minimum (min.), when tested as specified in 4.3.3.

3.3.2 pH.-The lead azide shall have a pH of 7.5 max, and 5.0 min, when tested as specified in 4.3.4.

3.3.3 Solubility in water.-The solubility of the lead azide in water shall be 1.0 percent max., when tested as specified in 4.3.5.

3.3.4 Matter insoluble in nitric acid.-The insoluble matter of the lead azide in nitric acid shall be 0.05 percent max. and none shall be retained on a U.S. Standard Sieve 230, when tested as specified in 4.3.6.

3.3.5 Iron and copper.-There shall be no more than trace quantities of iron and copper present when tested as specified in 4.3.7.

3.3.6 Chlorides, nitrates and acetates.-There shall be no more than trace quantities of chlorides, nitrates and acetates present, when tested as specified in 4.3.8.

3.3.7 Lead carboxymethyl cellulose.-The carboxymethyl cellulose when determined as the lead salt shall be 0.60 percent min. and 1.20 percent max., when tested as specified in 4.3.9.

3.3.8 Bulk density.-The bulk density shall be 1.1 gram (gm.) per milliliter (ml.) min. In addition, the maximum range of bulk densities between all batches in the lot (high value minus low value) shall not exceed 0.2 grams per milliliter (g/ml) when tested as specified in 4.3.10.

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3.4 Detonator function.-The detonator loaded with lead azide RD-1333 shall form a hole in the lead disc of 0.156 inch diameter, min., when tested as specified in 4.3.11.

3.5 Workmanship.-The lead azide RD-1333 shall be free from grease, wood slivers or chips, rubber or plastic particles, or any other foreign particles. During manufacture, care must be exercised to assure that the precipitation kettles are thoroughly cleaned before manufacture of each new batch. (Note: The cleaning may be mechanical (i.e., scouring with a wet sponge or a strong stream of water) or chemical (i.e., destroying the azides chemically, followed by thorough rinsing with water). However, the kettles must be cleaned chemically before preparation of every fourth batch if mechanical cleaning is chosen.) The solution of sodium carboxymethyl cellulose must be filtered prior to use in such a manner to preclude the presence of fibrous material. The lead acetate solution must be filtered until clear (i.e., no suspension of lead carbonate) and must be stored in such a manner as to preclude the formation of lead carbonate due to atmospheric carbon dioxide contamination.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection.-Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.1.1 Contractor quality assurance system.-If the contractor desires to utilize a quality assurance system which is at variance with the quality assurance provisions of 4.2 and 4.3 and other documents referenced herein, he shall submit a written description of the system to the Contracting Officer

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for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections to be performed, inspection stations, sampling procedures, methods of inspection (measuring and testing equipment) and provisions for control and disposition of non-conforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance required by the provisions of 4.2 and 4.3 and the other documents referenced herein. The contractor shall not be restricted to the inspection station or the method of inspection listed in this specification provided that an equivalent control is included in the approved quality assurance procedures. In cases of dispute as to whether certain procedures of the contractor's system provide equal assurance, the comparable procedure of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any change to the written procedure that affects the degree of assurance required by this specification or other documents referenced herein.

4.1.2 Submission of product.-At the time the completed lot of product is submitted to the Government for acceptance, the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product being submitted:

- a. A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.
- b. Quantity of product inspected.
- c. Results obtained for all inspection performed.
- d. Specification number and date, together with an identification and date of changes.
- e. Certificates of analysis on all material procured directly by the contractor including a statement that NaCMC has been used.
- f. Quantity of product in the lot.
- g. Date submitted.

The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

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4.1.3 First article inspection

4.1.3.1 Submission.-The contractor shall submit a first article quantity as designated by the Contracting Officer for evaluation in accordance with the provisions of 4.1.3.2 (see 6.2). The first article sample shall consist of 250 grams of material. The sample submitted shall have been produced by the contractor (or furnished by a supplier) using the same production processes, procedures, and equipment as will be used in fulfilling the contract. All materials, including packaging and packing, shall be obtained from the same sources of supply as will be used in regular production. The sample shall be accompanied by certificates of conformance. A first article quantity, or portion thereof, as directed by the Contracting Officer, shall also be submitted whenever there is a lapse in production for a period in excess of 90 days, or whenever a change occurs in manufacturing process, material used, drawing, specification or source of supply as to significantly affect product uniformity as determined by the Government. Prior to submission, the contractor shall inspect the sample to the degree necessary to assure that it conforms to the requirements of the contract and submit a record of this inspection with the sample. A sample containing known defects will not be submitted unless specifically authorized by the Contracting Officer.

4.1.3.2 Inspections to be performed.-The sample will be subjected by the Government to any or all of the examinations or tests specified in 4.2 and 4.3 of this specification and any or all requirements of the applicable drawings.

4.1.3.3 Rejection.-If the sample fails to comply with any of the applicable requirements, the first article quantity shall be rejected. The Government reserves the right to terminate its inspection upon any failure of a sample to comply with any of the stated requirements. In the event of rejection, the Government reserves the right to require the contractor to take corrective action and submit a new first article quantity or portion thereof. Until a first article quantity is accepted, the contractor is in no way authorized by the Government to resume regular production unless otherwise directed by the Contracting Officer.

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4.2 Inspection provisions

4.2.1 Lot formation.-A lot shall consist of one or more batches of lead azide RD-1333, produced by one manufacturer, under one continuous set of operating conditions, in accordance with the same specification or same specification revision. Furthermore, a new makeup of any one of the solutions (i.e., lead acetate, sodium azide or sodium CMC) constitutes the formation of a new lot. Each precipitation shall be a maximum of 10 pounds of lead azide RD-1333. A batch is that quantity which has been subjected to one physical mixing process to make it homogeneous. The batch size shall not exceed 15 pounds. There shall be no intermixing of batches. Each batch shall be identified with a batch identification number, name of manufacturer, date of manufacture as well as dry weight of lead azide contained therein.

4.2.2 Examination.-Inspection for Critical defects shall be 100 percent. Sampling plans and procedures for Major and Minor defects shall be in accordance with MIL-STD-105 except that continuous sampling plans in accordance with Standard MIL-STD-1235 may be used if approved by the procuring activity. Also, at the option of the procuring activity, AQL's and sampling plans may be applied to individual characteristics listed using an AQL of 0.65 percent for each Minor defect and an AQL of 0.40 percent for each Major defect.

4.2.2.1 Bag, cotton duck prior to closing (see 5.1).

Categories	Defects	Method of Inspection	Code No (see 6.4)
Critical:			
	1. Lead Azide insufficiently wet.	Visual	01001
Major: AQL 0.40 percent			
	101. Cloth cap pierced, torn or improperly positioned.....	Visual	01002
Minor: AQL 0.65 percent			
	201. Evidence of poor workmanship (see 3.5).....	Visual	01003

4.2.2.2 Bag, cotton duck (see 5.1)

Categories	Defects	Method of Inspection	Code No
Critical: None defined.			

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Major: AQL 0.40 percent
 101. Bag pierced or torn.....Visual 02001
 102. Bag improperly closed.....Visual 02002

Minor: None defined.

4.2.2.3 Bag, waterproof prior to closing (see 5.1)

Categories	Defects	Method of Inspection	Code No
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Critical: None defined.

Major: AQL 0.40 percent
 101. Cotton duck bag missing.....Visual 03001

Minor: None defined.

4.2.2.4 Bag, waterproof (see 5.1)

Categories	Defects	Method of Inspection	Code No
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Critical:
 1. Bag pierced or torn.....Visual 04001
 2. Bag improperly closed.....Visual 04002

Major: None defined.

Minor: None defined.

4.2.2.5 Container, prior to closing (see 5.1)

Categories	Defects	Method of Inspection	Code No
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Critical: None defined.

Major: AQL 0.65 percent
 101. Jute liner torn.....Visual 05001
 102. Sawdust insufficiently saturated.....Visual 05002
 103. Filled waterproof bag improperly surrounded by sawdust.....Visual 05003
 104. Container pierced, cut or damaged.....Visual 05004

Minor: None defined.

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4.2.2.6 Closed container (see 5.2)

Categories	Defects	Method of Inspection	Code No
Critical:			
1..	Marking misleading or unidentifiable.....	Visual	06001
Major:			
	AQL 0.40 percent		
101.	Container improperly closed or sealed.....	Visual- Manual	06002
Minor:			
	AQL 1.0 percent		
201.	DOD symbol misleading or unidentifiable.....	Visual	06003
202.	Bare spot on exterior other than slight scratches (applicable metal containers)...	Visual	06004

4.2.3 Testing (Note: To safely dispose of waste lead azide, add all azide residues or contamination into 50 times its weight of 15 percent ceric ammonium nitrate.)

4.2.3.1 Sampling.-The tests depicted in 4.3.1, 4.3.2 and 4.3.10 shall be performed on an individual specimen from each batch. If any of the samples fail to comply with the requirements specified herein, the batch shall be rejected. The tests depicted in 4.3.3 thru 4.3.9 and 4.3.11 shall be performed on a composite sample obtained by removing equal quantities of lead azide from each batch of lead azide in the lot and mixing to form a composite sample.

4.2.3.1.1 Preparation of the composite sample (see 4.3.3 thru 4.3.9 and 4.3.11).-An equal portion of lead azide RD-1333 shall be removed from each batch in the lot, by means of a horn spoon to form a sample of approximately 100 grams (dry weight). (Note: Samples may be taken from the agitated vessel before the lead azide is transferred from the vessel to the shipping bag). The sample shall be blended on a smooth surface by mixing with a horn spatula. Water shall be added in case the sample appears dry. The sample shall be spread out and divided into squares approximately 1/2 inch on a side, by means of the spatula, and small portions shall be taken from each square to make a retained sample of approximately 50 grams (dry weight). These portions, thoroughly wetted, shall be transferred to a smooth-necked bottle, tightly stoppered with a rubber stopper, and the stopper secured with adhesive tape. This retained sample bottle shall be so labeled that the batch from each lot from which the material was taken can be easily identified. The retained sample shall be held for possible future examination (six

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months minimum). The remaining portion of the sample shall be used for testing. This sample, thoroughly wetted, shall be transferred to a smoothnecked bottle tightly stoppered with a rubber stopper and the stopper secured with adhesive tape. The bottle shall be labeled to show the name of the material, manufacturer, plant, contract or order number, lot number and number of pounds in the lot. The tests specified in 4.3.3 thru 4.3.9 and 4.3.11 shall be performed on this sample. If the sample fails to comply with the requirements specified herein, the lot shall be rejected.

4.2.3.2 Detonator functioning (see 6.3).-A sample of fifty M47 detonators shall be loaded with lead azide RD-1333 and tested as specified in 4.3.11. If one detonator fails to comply with the requirements, a second sample of 50 detonators shall be loaded and tested. If two or more detonators of the combined samples fail to comply with the requirements, the lot of lead azide RD-1333 shall be rejected. If a dud (defined as failure to be initiated by action of the drop weight on the firing pin) occurs it shall not be considered as failure and a new detonator shall be substituted for each dud and tested.

4.3 Test methods and procedures.-The following tests shall be performed. (Note: Any deviations from the test and/or sampling procedures specified herein shall require prior approval by the procuring activity.)

4.3.1 Color - Major Defect - Defect Code 07001.-Determine the color of the lead azide by visual examination.

4.3.2 Form - Major Defect - Defect Code 08001.-Spread a thin layer of the sample on a glass slide and allow to dry at room temperature. Examine this material under a microscope using a magnification of approximately 150.

4.3.3 Purity.- Major Defect - Defect Code 09001.- (Note: Caution must be exercised. Evolution of hydrazoic acid which is toxic but odorless occurs. Therefore, operations where the lead azide is dissolved in an acid should be performed in a fume hood.)

4.3.3.1 Standard method

4.3.3.1.1 Apparatus.-Assemble the apparatus as shown on Figure 1. Insert the burette, containing water saturated with nitrogen, into one hole in the rubber stopper of the reaction flask. Add 90 ml. of 10 percent sodium hydroxide solution saturated with nitrogen to the 125 ml. carbon dioxide absorption flask. Fill the gas burette and leveling bulb with a 0.1 percent solution of Nacconol, or approved equivalent, saturated with nitrogen. Control the temperature of the system by circulating

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water by means of a pump, between the water reservoir which serves as a jacket for the reaction flask and the glass jacket of the gas burette. (Note: An approved equivalent type of apparatus may be used.) (See 6.5)

4.3.3.1.2 Determination.--Air dry part of the sample in a Buchner funnel and then heat in an oven maintained at 65-70 degrees Centigrade (degrees C.) to constant weight, but not more than 25 hours. (Note: An alcohol wash may be used to aid in drying. Transfer an accurately weighed portion of approximately 1.7 gm. of the dried sample to a glass vial shown on Figure 1. Add 3 ml. of water to the sample and place the vial containing the sample plus the water erect in the reaction flask containing 75 ml. of 15 percent ceric ammonium nitrate solution saturated with nitrogen. Connect the reaction flask to the apparatus (to insure that the reaction and absorption flasks are connected to the apparatus without air leaks, apply a coat of molten paraffin wax to all rubber-to-glass joints of these assemblies) without disturbing the position of the vial. Open stopcocks 1 and 2 to the atmosphere by adjusting them as shown by position A on Figure 1. Adjust the water level in the gas burette to zero with the aid of a leveling bulb. Allow the system to come to a temperature equilibrium by waiting 10 minutes after connecting the reaction flask. Read the temperature of the thermometer in the water jacket to 0.1 degree C. Turn stopcocks 1 and 2 to position B and shake the reaction flask so that the vial inside of it is upset and assumes a horizontal position at the bottom of the reaction flask. As the gas is evolved from the reaction mixture, lower the leveling bulb so that the liquid level in the leveling bulb is slightly below that in the gas burette. Gently agitate the reaction flask occasionally, to aid in completing the decomposition of the lead azide. When all the lead azide has decomposed, as indicated by the fact that gas bubbles are no longer forming in the mixture in the reaction flask, fill the flask with a measured volume of water from the water burette. Allow the temperature of the system to adjust itself to within 0.1 degree C. of its temperature at the beginning of the determination and then measure the volume of gas in the gas burette at the existing atmospheric pressure. Determine the atmospheric pressure to the nearest 0.1 mm. of mercury with the aid of a mercurial barometer having a brass scale. Correct the observed reading to 0 degrees C. Calculate the percent lead azide as follows:

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$$\text{Percent lead azide} = \frac{0.1558 (A-B) (C-D)}{(273 \text{ plus or minus } t) W}$$

where:

- A = ml. of gas measured in gas burette.
- B = ml. of water added to reaction flask.
- C = atmospheric pressure in mm. of mercury.
- D = vapor pressure of water in mm. of mercury at temperature t.
- W = weight of dry sample in gms.
- t = temperature of water in the jacket surrounding gas burette in degrees C.

4.3.3.2 Distillation titration method (alternative to the standard method.)

4.3.3.2.1 Apparatus (see Figure 2).--The apparatus consists of a heating mantle, a 125 ml. round bottom distilling flask, a three-way side arm adapter, a 50 ml. burette fitted with a number 5 size rubber stopper, water cooled condenser, adapter, and a 125 ml. Erlenmeyer flask. Standard taper ground glass joints are used to connect the distilling flask, side-arm adapter, condenser and adapter. The three way side-arm adapter has a female joint at its upper end to which the burette is fitted with the Number 5 rubber stopper. A thin film of silicone grease may be applied to all of the ground glass joints.

4.3.3.2.3 Solutions

- a. Ceric ammonium nitrate, 0.1 Normal (N). This solution is made up 2N with respect to perchloric acid.
- b. Sodium oxalate, 0.1N. The NBS oxidimetric standard is used.
- c. Perchloric acid, 3N.

4.3.3.2.3 Procedure.--Air dry a portion of the sample in a Buchner funnel and then heat in an oven maintained at 65-70 degrees C. until constant weight is obtained (See 4.3.3.1.2). Transfer an accurately weighed portion of from two to three milli-equivalents (0.2911 to 0.4366 gms.) of the dried sample to a small porcelain (or glass) crucible or boat. Cover the sample with water. To prevent foaming add a small amount of Dow Corning Anti-Foam AF Emulsion, or equivalent, to the reaction flask before the introduction of the sample. Place the sample in the distilling flask, using rubber-tipped forceps. Connect the ground glass joints of the apparatus. Connect the burette to the three-way side-arm adapter by means of the Number 5 rubber stopper. Transfer an accurately measured volume of about 40 to 50 ml. of 0.1N ceric ammonium nitrate to the Erlenmeyer flask in a position that the adapter from the condenser extends below the surface of the ceric ammonium nitrate. Fill the burette with 3N perchloric acid.

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Caution shall be exercised when adding perchloric acid to the distilling flask. Check to assure that all glass joints are sealed, open stopcock of burette and add 50 ml. of 3N perchloric acid. Distill the mixture in the distilling flask for approximately 12 minutes. Disconnect the adapter and rinse thoroughly with distilled water. Collect the rinse water in the receiver flask. Add one drop of 0.025N 5-Nitro-1,10 phenanthroline (nitro ferroin) to the receiver flask and titrate the excess cerate with standard 0.1N sodium oxalate to the nitro ferroin end point (denoted by a color change from red to pale greenish blue). Make a blank determination on the reagents and apply correction if necessary. Standardize the ceric ammonium nitrate solution with sodium oxalate. This standardization can be made by titrating 40.0 ml. of the cerate solution with the standard oxalate solution. Calculate the percent lead azide as follows:

$$\text{Percent lead azide} = \frac{N (B-A)}{W} 14.56$$

where:

A = volume of sodium oxalate.

N = normality of sodium oxalate, ml.

B = volume of sodium oxalate used in blank determination, ml.

W = weight of sample, gm.

4.3.4 pH - Major Defect - Defect Code 10001.-Determine the pH of a 10-gram specimen of lead azide in accordance with Method 103.1 of Military Standard MIL-STD-650.

4.3.5 Solubility in water - Major Defect - Defect Code 11001.-Mix the sample thoroughly, air-dry a portion on a Buchner funnel, and transfer approximately 5 gms. of the damp material to a tared, filtering crucible. Heat the crucible and contents in an oven maintained at 65-70 degrees C. to constant weight but not more than 25 hours, cool in a desiccator, and weigh. Wash the dry lead azide with five 10-ml. portions of distilled water at 0 degrees to 15 degrees C., allowing each portion to remain in contact with the lead azide for three minutes. Catch the filtrate in a clean filtering flask and retain for determination described in 4.3.8. Aspirate for five minutes. Dry the crucible and contents in an oven maintained at 65-70 degrees C., to constant weight but for no more than 25 hours, cool in a desiccator and weigh. Calculate the loss in weight of the lead azide to percent solubility of the sample in water, on a moisture-free basis.

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4.3.6 Matter insoluble in nitric acid - Major Defect - Defect Code 12001.-Mix the sample thoroughly, air-dry a portion on a Buchner funnel and transfer approximately 5 gms. of the damp material to a tared 150 ml. beaker. Heat the beaker and contents in an oven maintained at 65-70 degrees C. to constant weight but for not more than 25 hours, cool in a desiccator, and weigh. Add 100 ml. of dilute nitric acid (1:4) to the beaker, and warm beaker with contents on a hot water bath. Allow any insoluble matter present to settle and decant supernatant liquid through a tared, filtering crucible, catching the filtrate in a clean filtering flask. Repeat the previous operation (adding 100 ml. of dilute nitric acid, warming on hot bath and filtering) two additional times. Transfer quantitatively the residue in the beaker to the tared filtering crucible with the aid of distilled water. Remove tared, filtering crucible from filtering flask and attach to another filtering flask (save filtrate for determination described in 4.3.7). Wash contents of crucible with five 20 ml. portions of five percent sodium hydroxide, allowing each portion of the sodium hydroxide to remain in contact with the residue for 1 minute. Wash crucible and contents with three 20 ml. portions of cold distilled water. Aspirate for five minutes. Dry the crucible and contents in an oven maintained at 105° plus or minus 5°C. for 1 hour, cool in a desiccator and weigh. Calculate the gain in weight of the crucible to percent insoluble in nitric acid. (Note: Caution must be exercised. In this test the lead azide is dissolved in dilute nitric acid, a process which gives rise to the evolution of hydrazoic acid gas which is toxic but odorless. Therefore, this operation should be done in a fume hood.

4.3.7 Iron and copper**4.3.7.1 Solutions for iron determination:**

a. Ammonium mercaptoacetate (thioglycollic acid).-Neutralize a solution of 10 grams of mercaptoacetic acid in 100 ml. of distilled water with ammonium hydroxide using litmus paper as the external indicator.

b. Standard iron solution, Dissolve 0.175 g. of ferrous ammonium sulfate in distilled water, add 2ml. of 4N sulfuric acid and make up to 1 liter. One ml. of this solution, based on the 2.5 gram sample weight, is equivalent to 0.001 percent iron.

4.3.7.2 Sample Preparation.-Warm the filtrate reserved from 4.3.6 and slowly add 40 ml. of 4N sulfuric acid. Allow the solution to digest on a steam bath for 1 hour. Filter the solution through a filtering crucible, catching the filtrate in a clean filtering flask. Transfer the filtrate to a clean 250 ml. beaker

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and evaporate the contents to fuming on a hot plate. Continue the evaporation until approximately 1 ml. remains. Allow the residue to cool and add about 30 ml. of cold water. Filter off any further precipitate and make the filtrate up to 50 ml. Use for the following iron and copper determinations:

4.3.7.3 Iron procedure - Major Defect - Defect Code 13001.-Add 2 ml. of 20 percent citric acid to 25 ml. of the filtrate obtained in 4.3.7.2 and neutralize with dilute ammonium hydroxide (1:4) using litmus paper as an external indicator. Add 2 ml. of 10 percent ammonium mercaptoacetate (thioglycollic acid), 10 ml. of 4N ammonium hydroxide and make up to 50 ml. with distilled water. Treat a one (1) ml. sample of the standard similarly. Compare visually the colors produced. The color of the determined iron should be the same or lighter than that of the standard.

4.3.7.4 Solutions for copper determination:

a. Rubeanic acid - Dissolve 0.1 gm. of rubeanic acid in 100 ml. of methyl alcohol.

b. Standard copper solution - Dissolve 0.0393 gm. of copper sulfate in distilled water and make up to 1 liter. One ml. of this solution, based on the one gram sample weight, is equivalent to 0.001 percent copper.

4.3.7.5 Copper procedure - Major Defect - Defect Code 14001.-Neutralize 10 ml. of the filtrate obtained in 4.3.7.2 with ammonium hydroxide using litmus paper as an external indicator. Then add 2 ml. of glacial-acetic acid, 5 ml. of 20 percent ammonium acetate solution and 0.5 ml. of 0.1 percent rubeanic acid. Make up to 50 ml. with distilled water. Treat one ml. of the standard copper solution similarly. Compare visually the colors produced. The color of the determined copper should be the same or lighter than that of the standard.

4.3.8 Chloride, nitrates and acetates.-Divide the filtrate obtained in 4.3.5 into two equal portions. Retain second portion for the acetate determination in 4.3.8.6. Pass hydrogen sulfide through the first portion for a few minutes and allow the beaker to stand on a steam bath to remove all the hydrogen sulfide and to coagulate the precipitate. Filter off the precipitate and make the volume of the filtrate up to 50 ml. Divide filtrate into two equal 25 ml. portions which will be used for chloride and nitrate determination in 4.3.8.2 and 4.3.8.4.

4.3.8.1 Solution for chloride determination:

a. Nitric acid 70 percent

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b. Silver nitrate - 0.1N, dissolve 1.69 gms. of silver nitrate in 100 ml. distilled water.

c. Standard chloride solution.-Dissolve 0.085 g. of potassium chloride in 500 ml. distilled water.

4.3.8.2 Chloride procedure - Major Defect - Defect Code 15001.-Transfer first portion of filtrate obtained in 4.3.8 to a Nessler tube. Add 1 ml. concentrated nitric acid and 1 ml. 0.1N silver nitrate solution. Compare the turbidity with that of a standard, equivalent to 0.02 percent lead chloride, in a second Nessler tube. Place 1 ml. of the standard chloride solution in a Nessler tube, add 1 ml. of 0.1N silver nitrate and make up to 50 ml. The turbidity present in the chloride determination should be the same or less than that of the standard.

4.3.8.3 Solutions for nitrate determination:

a. Diphenylamine.-Dissolve 0.5 gm. of diphenylamine in 50 ml. of sulfuric acid.

b. Standard nitrate solution.-Dissolve 0.125 gms. lead nitrate in 500 ml. of distilled water.

4.3.8.4 Nitrate procedure - Major Defect - Defect Code 16001.-Transfer second portion of filtrate obtained in 4.3.8 to a 100 ml beaker and evaporate on a steam bath to about 2 ml. Transfer, quantitatively, to a test tube, add 2 ml. conc. sulfuric acid and 1 drop diphenylamine solution. The color obtained shall be equal or less than the color obtained with that of a standard (i.e., pipet 2 ml. of standard nitrate solution into a test tube, add 2 ml. conc. sulfuric acid and 1 drop of diphenylamine solution).

4.3.8.5 Solutions for acetate determination:

a. Iodine solution A - Dissolve 1.27 gms. of iodine and 1.6 gms. of potassium iodide in 100 ml. of distilled water.

b. Iodine solution B - Dissolve 0.127 gms. of iodine and 0.16 gms. of potassium iodide in 100 ml. distilled water.

c. Lanthanum nitrate solution - Dissolve 5 gms. of lanthanum nitrate in 100 ml. distilled water.

d. Standard acetate solution - Dissolve 0.05 gms. of sodium acetate in 500 ml. of distilled water.

4.3.8.6 Acetate procedure - Major Defect - Defect Code 17001.-add 1 ml. of iodine solution A to the filtrate obtained in 4.3.8 and evaporate the solution to about 2 ml. on a steam bath. Cool the solution and quantitatively transfer it into a graduated

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test tube which has a one ml. marking. Continue the evaporation in the test tube until the volume has been reduced to 1 ml. Cool and add 3 drops of iodine solution B, 3 drops of 5 percent lanthanum nitrate and 4 drops of 0.5N ammonium hydroxide in that order, shaking after each addition. Bring the solution to a boil and allow it to stand for 10 minutes. Perform a comparative test starting with 25 ml. of standard sodium acetate solution using the procedure of adding iodine solution A, evaporation, adding iodine solution B, evaporation, etc. The color produced by the test should be no greater than that produced by the standard.

4.3.9 Lead carboxymethyl cellulose - Major Defect - Code 18001.- Transfer 2.00 gms. of a dried sample of the lead azide RD-1333 to a 250 ml. beaker. Add 75 ml. of glacial acetic acid to the beaker and place beaker with contents on a steam bath. Swirl gently for a few minutes. Allow the beaker to stand on the steam bath, covered with a watch glass, for 30 minutes. Filter hot through a tared sintered glass crucible. Use an additional 50 ml. of hot glacial acetic acid to rinse beaker and crucible. Treat residue in crucible with three 20 ml. portions of cold 10 percent lead acetate solution, by filling the crucible with the lead acetate solution and applying gentle suction. Wash thoroughly with hot distilled water, dry at 105 plus or minus 5° C. for 1 hour, cool in a desiccator and weigh. Calculate the percentage of lead carboxymethyl cellulose as follows:

$$\text{Percent lead carboxymethyl cellulose} = \frac{(A-B) \times 100}{C}$$

where:

- A = weight of crucible plus residue
- B = weight of crucible
- C = dry weight of sample

CAUTION: In this test there is an evolution of hydrazoic acid gas which is very toxic. The determination should be performed in a fume hood.

4.3.10 Bulk density

4.3.10.1 Preparation of specimen - Major Defect - Defect Code 19001.-

4.3.10.1.1 Applicable to dry lead azide.-Transfer a portion of approximately 4 gms, to a drying dish. Place the dish and contents into an oven, maintained at 65-70 degrees C., for 1 hour, and then cool in a desiccator to room temperature.

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4.3.10.1.2 Applicable to wet lead azide.-Transfer a portion of approximately 4 gm to a 50 ml. beaker. Decant off the excess water and add 20 to 30 ml. of 95 percent ethyl alcohol. Gently swirl the beaker and contents for a few minutes and then decant off the alcohol. Repeat the procedure of adding alcohol, swirling and decanting once more. Then add 20 to 30 ml. of anhydrous ethyl ether and swirl the beaker and contents for a few minutes. Allow the lead azide to settle and decant the ether off. Air dry the sample until no ether odor is detected. Place the beaker and contents in an oven, maintained at 65-70 degrees C., for 1 hour, and then cool in a desiccator to room temperature.

4.3.10.2 Aluminum charge plate.-The aluminum charge plate shall be 3 inches long, 2 inches wide with a single cavity near the center which has a volume of about 0.1467 ml. The cavity shall be a frustrum with a top diameter of 0.230 inch, a bottom diameter of 0.177 inch and a height of 0.278 inch. Determine the volume of the cavity by dividing the mercury used to fill the cavity by the density of mercury at the working temperature.

4.3.10.3 Procedure.- (CAUTION: Perform this test behind a properly barricaded and grounded area and limit the quantity of lead azide in the area to only that which is going to be tested.) Place the aluminum charge plate on a piece of glazed paper and fill the cavity with lead azide by means of a plastic dipper. Level off the excess lead azide, with the aid of a cardboard spatula, so that it is even with the surface of the plate. Take care that the excess azide remains on the plate thereby allowing none of the sample to fall onto the piece of glazed paper. Lift the plate from the glazed paper, which effects a transferral of the lead azide to the glazed paper. Transfer this specimen to a tared weighing pan. Repeat the above procedure until five charges are collected in the weighing pan. Weigh the pan and determine the bulk density by the following formula:

$$\text{Bulk density, gm./ml.} = \frac{A}{B}$$

where:

A = weight of the 5 charges.

B = five times the volume of the charge plate cavity.

4.3.11 Detonator functioning - Major Defect - Defect Code 20001.-

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4.3.11.1 Standard method.-Fifty M47 detonators shall be loaded in accordance with drawing 73-2-335 except that 25 milligrams of RD-1333 lead azide shall be used for the intermediate charge and 70 milligrams of RDX (min.) shall be used for the base charge. The detonators shall be tested using the adapter shown on Figure 3 and 3.95 ounce steel ball, 1 and 3/16 inch in diameter, which is dropped on the firing pin from a height of 3 inches.

4.3.11.2 Alternate method.-The M47 detonator shell, containing the specified copper disc which rests on the flange of the shell, shall be inserted in an aluminum or steel loading block (design on a loading block optional depending on type of press to be used, etc.) and shall be loaded with 75 to 80 milligrams of RDX. The powder shall be pressed at approximately 100 pounds with flat faced aluminum or steel pin having a diameter of 0.123-0.125 inches. Then 25 milligrams of RD-1333 lead azide shall be loaded into the shell and pressed in the same manner as the RDX load.

The loaded detonator shall be removed from the loading block and then placed in the two-diameter holding sleeve. The unit shall then be positioned in the holding block. This test stage arrangement shall be so arranged that the bottom of the detonator and the two-diameter holding sleeve rests on the lead test plate. The lead test plate shall have a thickness of 0.1345 inches plus or minus 0.015 inch. Also a square plate approximately 1 inch by 1 inch shall be used. A piece of Orange Wax Clover, safety fuse approximately four inches long shall be fully inserted into the top end of the two diameter holding sleeve and then the complete detonator test assembly shall be made ready for firing. The test assembly shall be placed in an appropriate shield and the fuse shall be ignited with a match.

Any "dud" encountered in this test where the lead azide load is not ignited by the safety fuse shall be discounted and another detonator shall be loaded as a replacement. The criteria for proper lead azide performance shall be the initiation of the RDX load so as to produce the specific hole in the lead test plate. Ignition of the lead azide by the fuze is not a criterion for proper lead azide performance.

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging - Level C.-Discharge the wet washed azide from the kettle into a duck cloth bag (4 ounces or heavier) or into a diaper cloth approximately 30 inches square.

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(NOTE: This discharge from the kettle to the cloth may be accomplished by sluicing the lead azide from the tilted kettle with a low pressure water stream.) If a diaper cloth is used, form the cloth into a bag by gathering the edges of the cloth. Bind securely the top of this bag using heavy cotton cord. Connect an identification tag (made from polyethylene or rubber) to this cord (see 5.3). Each bag shall contain not more than 15 pounds (dry weight) of lead azide (a sample batch). Place up to three diaper bags into a 24 inch by 24 inch eight ounce cotton duck bag. Securely tie the mouths of these duck bags with heavy cotton cord. The material used (diaper cloth and duck cloth) shall not be impregnated with resinous materials. CAUTION: The lead azide shall be wet with a solution of denatured alcohol and water containing not less than 50 percent of ethyl alcohol. (NOTE: SD-1 alcohol may be used.)

5.2 Packing - Level C.-Containers complying with Specification 5 and 5B (metal barrels or drums), 17H (metal drums, single trip) or 10B (wooden barrels or kegs) of Regulation for Transportation of Explosives and Other Dangerous Articles, etc., shall be used for packing the bags described in 5.1. The packing shall be accomplished as follows:

Line the drum or barrel with a cloth drum lining made of Osnaburg or Jute material. Place several inches of clean sawdust in the bottom of the liner. Place two 8-mil polyethylene bags, one inside the other, standing upright on this bed of sawdust in the Osnaburg liner. Place the cotton duck bags within the double polyethylene bags inside the drum. No more than 153 pounds (dry weight) of lead azide shall be placed in any single drum. Fill the polyethylene bags completely with a solution of denatured alcohol/water (NOTE: SD-1 alcohol may be used) to obtain a resultant liquid which contains at least 50 percent alcohol. Seal the polyethylene bags by securely wrapping with plastic electrical tape. Fill the free space in the drum between the cloth liner and polyethylene bags with not less than 3 inches of well-packed sawdust saturated with 50/50 mixture of alcohol and water. Sew the outer bag to prevent escape of sawdust. Close and seal the drum or barrel.

5.3 Marking.-Marking to insure safe handling shall conform to Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc. In addition to any special marking required by the procuring activity, shipments shall be marked in accordance with Standard MIL-STD-129. Each bag shall be marked with batch identification numbers, name of manufacturer, date of manufacture and weight of dry lead azide contained therein.

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6. NOTES

6.1 Intended use.-The material covered by this specification is intended for use in the manufacture of detonators, fuzes, and priming compositions.

6.2 Ordering data.-Procurement documents should specify the title, number and date of this specification, and provisions for submission of first article samples.

6.3 Detonator metal parts required for this test will be furnished by the Contracting Officer upon request.

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

6.5 The description of apparatus and method submitted by Olin Industries, East Alton, Illinois on 7 October 1965, is an approved equivalent.

Custodian:
Army-MU

Preparing activity:
Army-MU

Project Number; 1376-A-020

Mel - L - 46225C

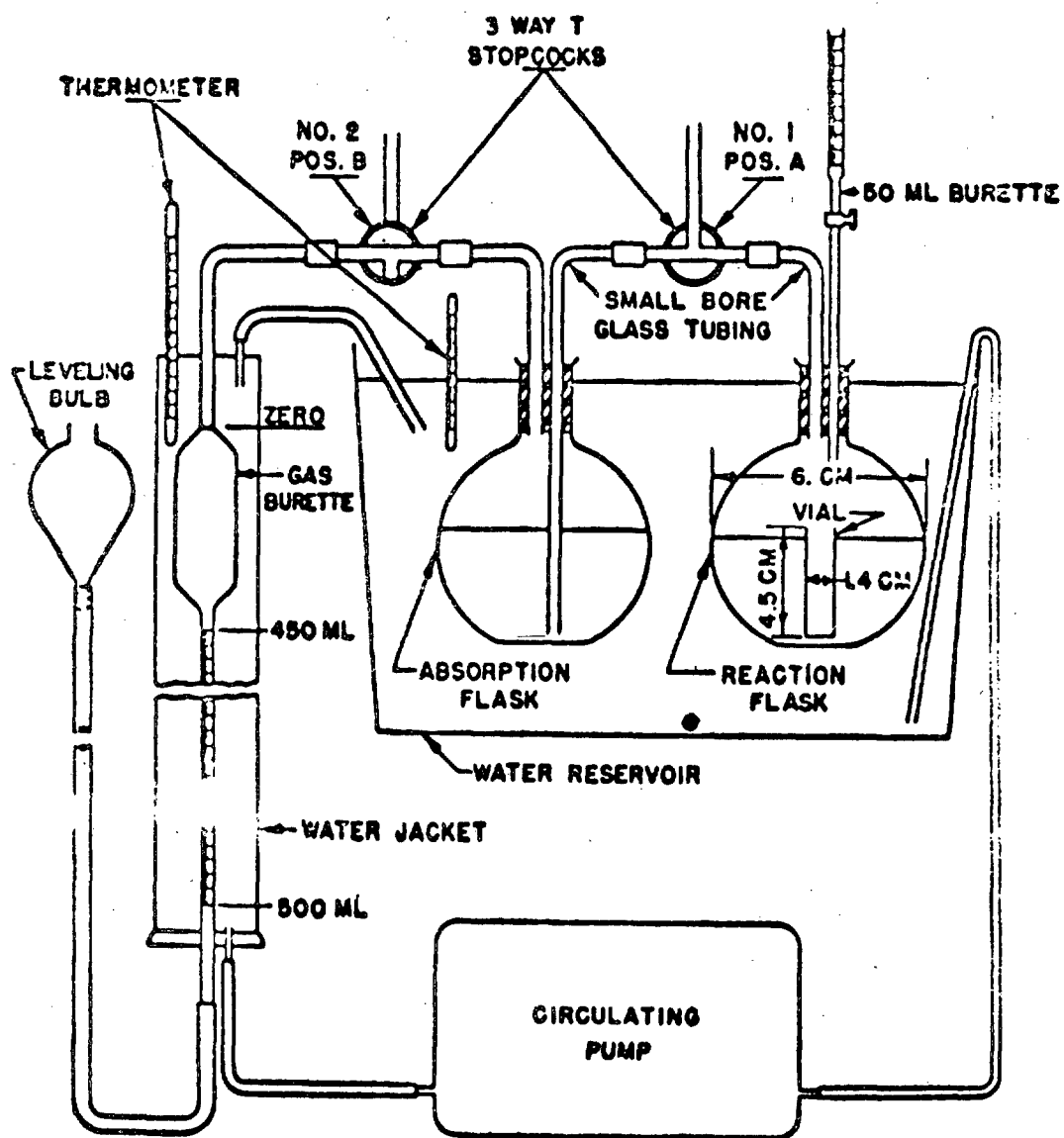
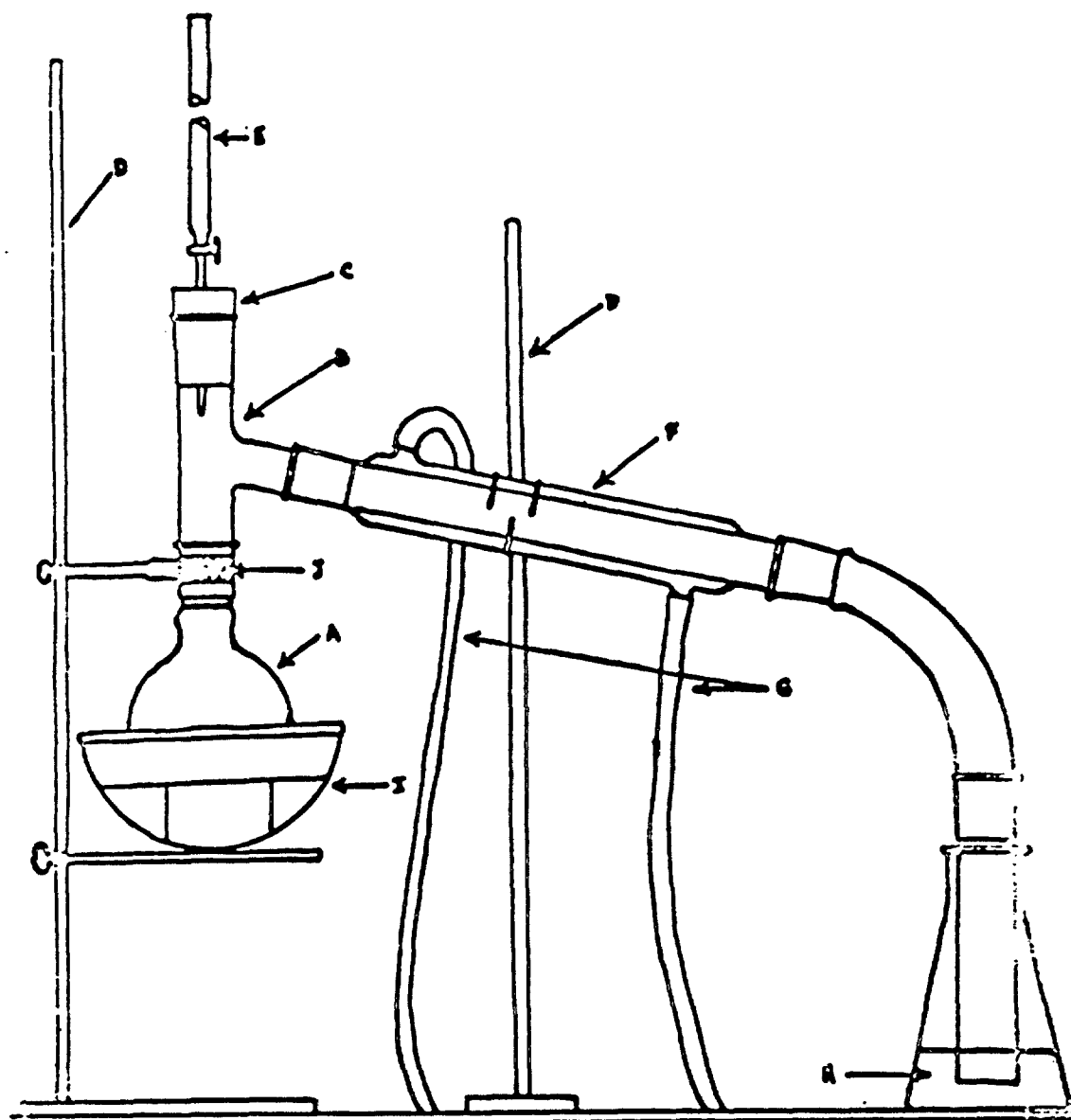


FIGURE 1.—Equipment assembly.

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A - Distilling Flask

B - Side-Armed Adapter

C - Rubber Stopper (25)

D - Clamp Stands

E - Burette

F - Condenser

G - Rubber Tubing

H - Cerio Ammonium Nitrate

I - Heating Mantle

J - Clamp

FIGURE 2

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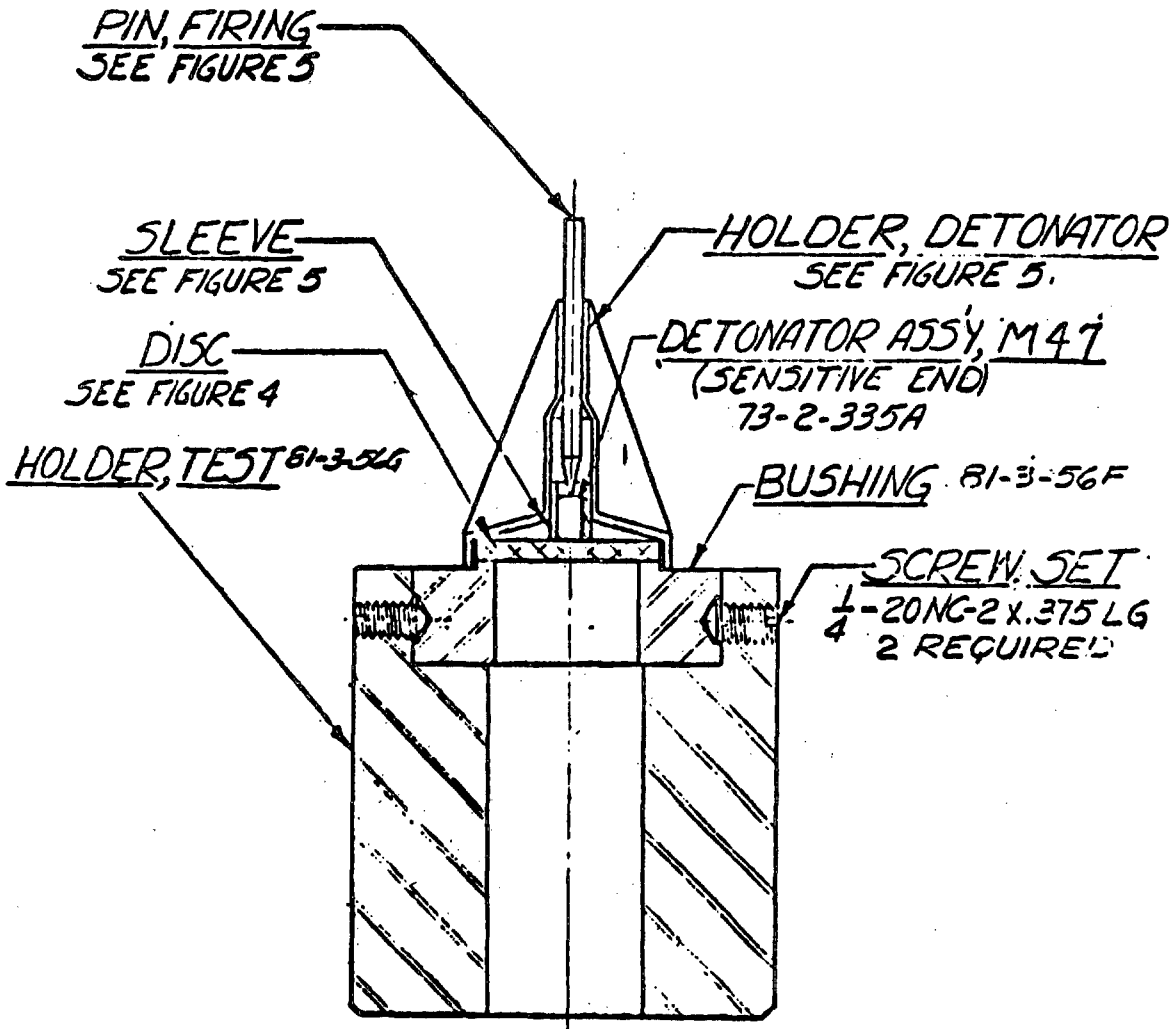
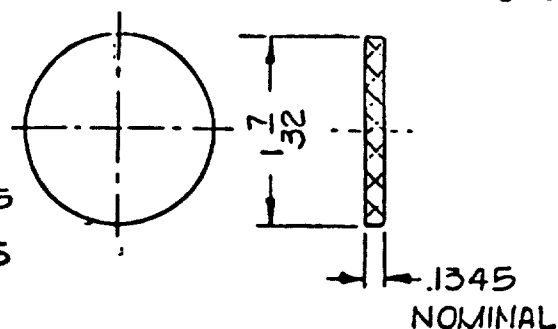
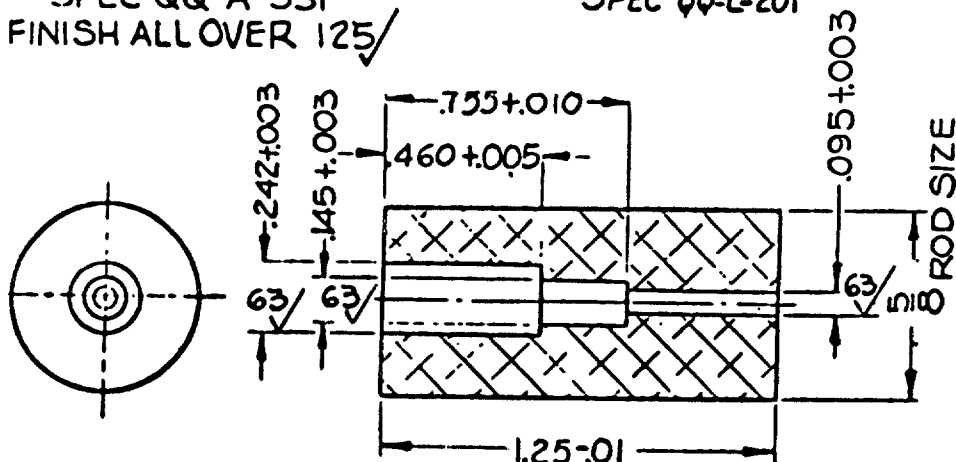


FIGURE 3

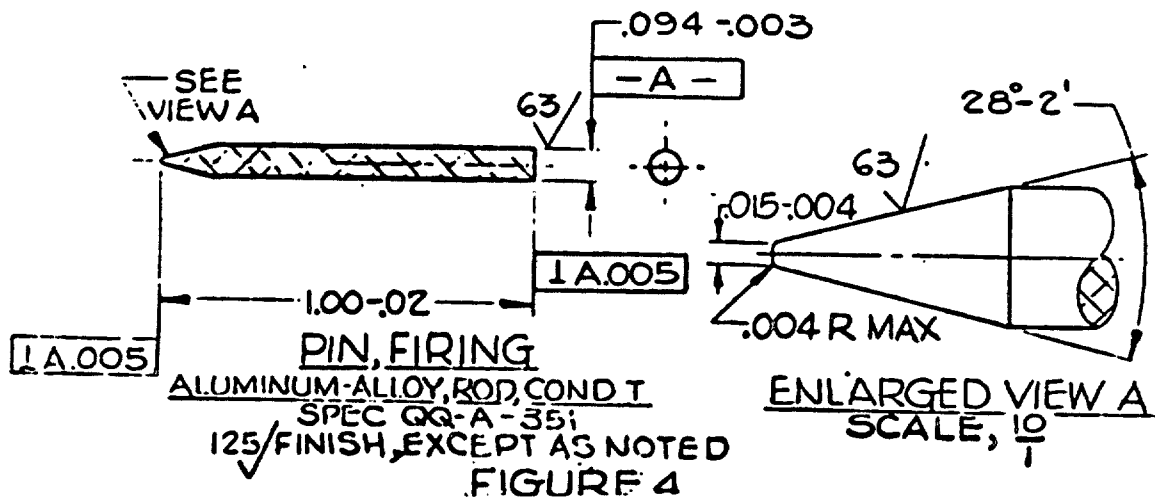


DISC

LEAD; SHEET, GRADE B, 8LB
SPEC 00-L-201



ALUMINUM-ALLOY ROD COND'T, SPEC. QQ-A-351
125/ALL OVER, EXCEPT AS NOTED



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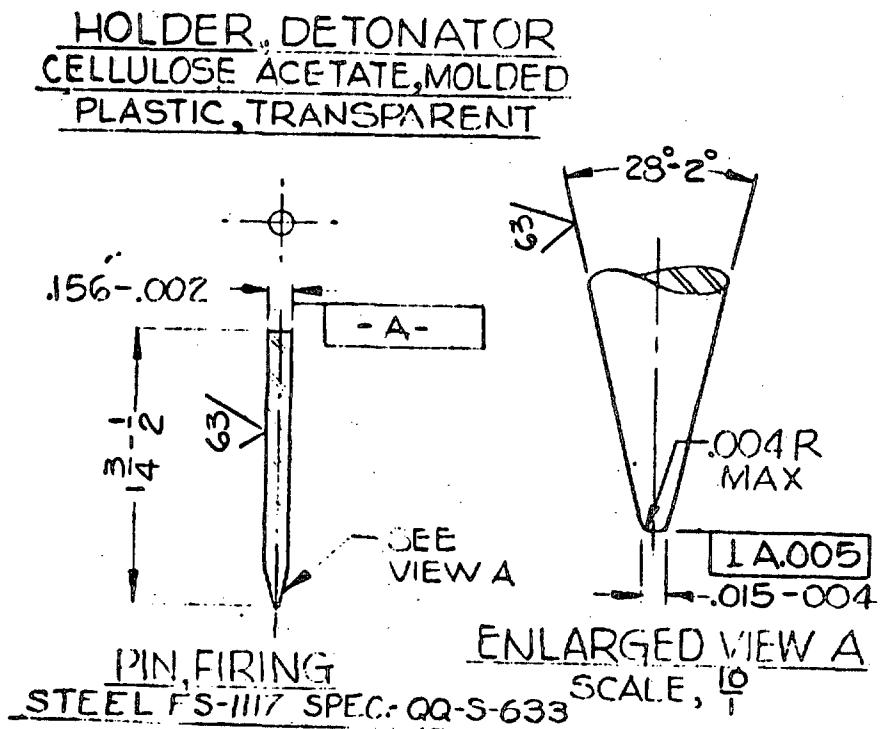
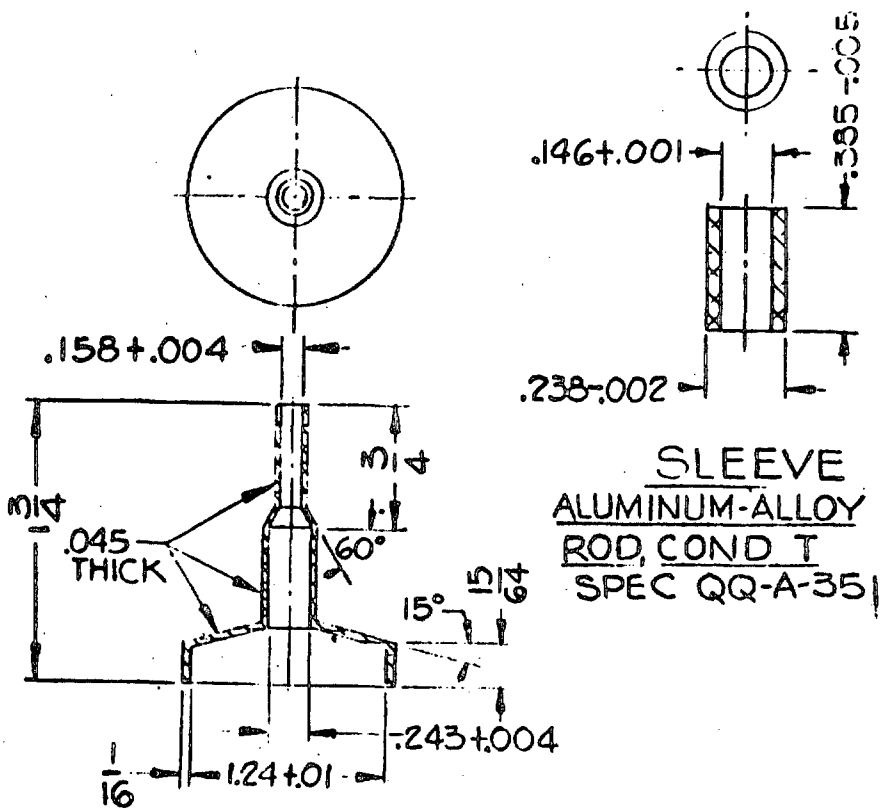


FIGURE 5

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M-47846/2	April 1956	PICATINNY ARSENAL	ORDNANCE CORPS
	Lead Azide		RD1333
	Sample No. 271-242		150X

Figure 6

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