

MIL-R -13742 (Ord)  
 3 November 1954  
SUPERSEDING  
 PA-PD-527  
 30 June 1954

## MILITARY SPECIFICATION

### RDX, DESENSITIZED

#### 1. SCOPE

1.1 This specification covers desensitized RDX intended for use as a component of Military dynamite.

#### 2. APPLICABLE DOCUMENTS

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

#### SPECIFICATIONS

##### MILITARY

MIL-L-2104	-Lubricating Oil, Internal-Combustion Engine, Heavy Duty
MIL-G-2550	-General Specification for Ammunition Except Small Arms Ammunition
MIL-R-398	-RDX

#### STANDARDS

##### MILITARY

MIL-STD-129	-Marking For Shipment and Storage.
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#### DRAWINGS

##### ORDNANCE CORPS

76-1-1405	-Box, Packing for High Explosives, Assembly, Details, Packing and Marking
76-1-1406	-Carton, Packing, Reusable - Collapsible for High Explosives. Assembly, Details, Packing and Marking
76-1-1407	-Carton, Packing, Reusable - Collapsible, Alternative for High Explosives, Assembly, Details, Packing and Marking

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## PUBLICATIONS

## ORDNANCE CORPS

ORD-SIP-M12-210-RDX, Desensitized

(Copies of specifications, standards, drawings, and publications required by the contractor 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 document forms a part of this specification. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

INTERSTATE COMMERCE COMMISSION  
49 CFR 71-78 - 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-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 Material. - The desensitized RDX shall be a thoroughly homogeneous mixture of RDX, complying with Specification MIL-R-398, Type II, Class 1, incorporated with Grade 10 engine oil, complying with Specification MIL-L-2104, to form a light yellow granular mixture.

3.2 Moisture. - Maximum, 0.10 percent, when determined as specified in 4.4.

3.3 Composition. - The proportions of RDX and engine oil in the desensitized RDX shall be as follows, when determined as specified in 4.5:

RDX	-	96.0	±	0.5 percent
Engine oil	-	4.0	±	0.5 percent

3.4 Acidity. - Maximum, 0.03 percent as acetic acid, when determined as specified in 4.6.

3.5 Inorganic insoluble matter. - Maximum, 0.05 percent, when determined as specified in 4.7.

3.6 Workmanship. - The desensitized RDX shall be free from foreign matter and visible impurities.

#### 4. QUALITY ASSURANCE PROVISIONS

4.1 Lot.--A lot shall consist of one or more batches of material subjected to the same processing operations and conditions. Each lot shall contain RDX from not more than one manufacturer.

4.2 Sampling.--Each sample selected shall consist of approximately 8 ounces of desensitized RDX. Number of samples, acceptance and rejection criteria, used for determination of lot acceptance for tests as specified herein shall be in accordance with Standard Inspection Procedure ORD-SIP-M12-210.

4.3 Inspection.--Inspection shall be as specified in Specification MIL-G-2550.

4.4 Moisture.--

4.4.1 Apparatus.--

4.4.1.1 Titration assembly.--The apparatus shown in figures 1, 2, and 3 shall be used. The rubber stopper shall be fitted with the electrodes, buret tips, and air vent to a drying tube, and placed in the iron ring. The sample port hole shall be closed with a glass stopper and the iron ring shall be fastened to a ring stand. The ring with stopper shall remain fixed throughout the determination. The titration vessel shall be fitted onto the stopper, and the magnetic stirrer placed beneath the beaker to serve as a support as well as a stirring mechanism.

4.4.1.2 Titration vessel.--A 200-ml tall form beaker, without pouring lip, as shown in figure 2, has been found satisfactory. The dried beaker shall be taken from the drying oven and immediately connected to the stopper in the titration assembly. As the beaker cools, dry air will be drawn in through the drying tube at the vent. A minimum of four dry beakers will be required, one for each of the following determinations:

- a. to determine "R" (see 4.4.3.1).
- b. to determine "F" (see 4.4.3.2).
- c. to titrate sample (see 4.4.4).
- d. to titrate blank (see 4.4.4).

4.4.1.3 Buret assembly.--Two all-glass buret assemblies (one for Karl Fischer reagent and one for water in methanol solution) to permit introduction of the reagent into the titration vessel (200-ml beaker, figure 2) by gravity from a reservoir without exposure to atmospheric moisture will be required. The Machlett automatic burets have been found satisfactory. The reservoirs connected to the burets with all glass tubing shall be protected against atmospheric moisture by drying tubes and protected from light with black paint or dark paper on the outside of the reservoir bottles.

4.4.1.4 Electrodes.--Two platinum wire electrodes, 0.016 inch diameter, sealed-in-glass with mercury connections, shall be used.

4.4.1.5 Timer.--Any interval timer which can measure time to the nearest second and can record minutes shall be used.

4.4.2 Reagents and solutions.--

4.4.2.1 Karl Fischer reagent.--To make 1000 ml of the reagent, 264 grams of anhydrous pyridine of highest purity shall be placed in a 1000-ml Florence flask, and 61 grams of liquid SO<sub>2</sub> shall be added by inverting the cylinder and allowing the SO<sub>2</sub> to run through a glass tube into the pyridine in the flask on a balance pan. A 644-ml portion of anhydrous methanol (not more than 0.02 percent moisture) shall be added. A CaCl<sub>2</sub> drying tube shall be attached and the flask chilled in

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an ice bath. When thoroughly chilled, 65 grams of C.P. resublimed iodine shall be added at one time. The solution shall be kept chilled and agitated by swirling until the iodine has dissolved completely. Due to its rapid initial deterioration, the reagent should not be used for 24 hours. One ml is equivalent to approximately 0.0025 gram of water. Alternatively, commercially available Karl Fischer reagent may be used.

4.4.2.2 Standard water-in-methanol solution.-To a previously dried 1000-ml volumetric flask, 2 ml of water shall be pipetted, immediately diluted to the mark with anhydrous methanol, stoppered and mixed thoroughly.

4.4.2.3 Special solvent.-Equal volumes of anhydrous methanol and carbon tetrachloride shall be mixed. If necessary, the solvents may be dried by distillation.

4.4.2.4 Sodium tartrate dihydrate.-The water content of this material shall be established by determining the loss in weight on drying at 150°C for 3 hours. The water content of this material should be 15.66 ± 0.05 percent. If not, another fresh bottle of the material shall be used.

4.4.3 Standardization of solutions.-

4.4.3.1 Karl Fischer reagent.-A 100-ml portion of the special solvent shall be transferred to one of the dry 200-ml beakers (see figure 2) through the sample porthole of the rubber stopper. A slight excess of Karl Fischer reagent shall be added and titrated with standard water-in-methanol solution to the preliminary end point exactly as specified in 4.4.4. Immediately upon reaching the preliminary end point, the stop watch shall be released and buret readings taken. Approximately 10 ml of the reagent, accurately measured, shall be added and the solution shall be titrated immediately with standard water-in-methanol solution. These operations shall be controlled to reach the second end point within 3.5 minutes plus or minus 15 seconds from the preliminary end point. This second end point may be used as the preliminary end point for a check standardization. In this case, the stop watch shall be returned to zero and released as soon as the second end point has been reached, and the titration for the third end point shall be completed within an additional 3.5 minutes. The ml of standard water-in-methanol solution per ml of Karl Fischer reagent shall be calculated. This is the ratio "R" referred to in 4.4.3.2 in the calculation of "F". Check determinations should agree within 0.010. Because the ratio is constantly changing due to deterioration of the reagent, the standardization shall be made at least once a day, more often for the most accurate work.

4.4.3.2 Standard water-in-methanol solution.-A 100-ml portion of special solvent shall be transferred to another one of the dry 200-ml beakers through the sample porthole of the rubber stopper, and titrated with Karl Fischer reagent and standard water-in-methanol solution to the preliminary end point exactly as specified in 4.4.4. The stop watch shall be released and buret readings taken immediately. From a glass stoppered weighing bottle, about 0.5 grams of sodium tartrate dihydrate weighed by difference to the nearest milligram,

shall be quickly introduced. A slight excess of Karl Fischer reagent shall be added and back-titrated with standard water-in-methanol solution to the final exact end point within 3.5 minutes. The titration shall be repeated at least once and the standardization factor "F" calculated as follows:

$$F = \frac{0.1566W}{VR-S}$$

where:

F = grams of water per ml of standard water-in-methanol solution

W = grams of sodium tartrate dihydrate

V = ml Karl Fischer reagent used for titration

R = ml standard water-in-methanol solution per ml Karl Fischer reagent

S = ml standard water-in-methanol solution for back-titration

4.4.4 Procedure. -A 100-ml portion of the special solvent shall be transferred through the sample port-hole into the dry beaker of the titration assembly containing a plastic coated magnet, and stoppered immediately. The platinum electrodes shall be kept covered at all times, the magnetic stirrer shall be started and the water in this solvent titrated by adding a slight excess of the standardized Karl Fischer reagent to produce a reddish-brown color which persists for at least half a minute. The variable resistance should be adjusted at this point so that, with the excess Fischer reagent present, the needle of the micro-ammeter (see figure 1) is at the upper end of the scale. The solution shall be titrated slowly with the water-in-methanol solution until the exact preliminary end point is reached. The end point shall be defined as the point at which one drop of the standard water-in-methanol solution causes the micro-ammeter needle to waver and then slowly drift from the upper end of the scale toward the lower end is not less than 15 seconds. When the preliminary end point is reached the stopwatch shall be released. All succeeding operations shall be completed within  $18.5 \pm 0.25$  minutes. An 8-to 10-gm portion of the sample, weighed by difference from a glass-stoppered weighing bottle shall be introduced quickly into the beaker through the sample porthole of the rubber stopper. Loss of 0.05 gm of the sample has no appreciable effect on the accuracy of the determination. The porthole shall be closed immediately and the contents of the beaker stirred for 15 minutes by means of the magnetic stirrer. Then a slight excess of the standardized Fischer reagent shall be added. This causes the needle of the micro-ammeter to return to the upper part of the scale. The solution shall be carefully back-titrated with standard water-in-methanol solution to reach the exact and final end point in  $18.5 \pm 0.25$  minutes for the time the preliminary end point was reached. The final end point shall be obtained in the same manner as the preliminary end point (defined above) was obtained. The volume of the back-titration should be less than 1 ml. A blank determination shall be conducted using 100 ml of the special solvent only by titrating to the preliminary end point, stirring for 15 minutes, and then completing the titration to the exact final end point  $18.5 \pm 0.25$  minutes as specified above for the sample. The percent water in the sample shall be

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calculated as follows:

$$\text{Percent water} = \frac{F((VR-S)-(V'R - S'))}{W} 100$$

where:

- F = gms of water per ml of standard water-in-methanol solution as determined in 4.4.3.2.  
 V = ml of Karl Fischer reagent added to sample  
 V' = ml of Karl Fischer reagent added to blank  
 R = ml of standard water-in-methanol solution per ml of Karl Fischer reagent  
 S = ml of standard water-in-methanol solution for back-titration of sample  
 S' = ml of standard water-in-methanol solution for back-titration of blank  
 W = gms of sample

(Note: The presence of moisture in the solution in which the platinum electrodes are immersed causes the electrodes to polarize which stops the flow of current and causes the needle of the micro-ammeter to move to the lower end of the scale. The presence of an excess of Karl Fischer reagent depolarizes the electrodes which permits current to flow and causes the needle to move to the upper end of the scale.)

#### 4.5 Determination of composition.

4.5.1 RDX content.-An accurately weighed portion of approximately 2 grams of the sample shall be transferred to a 100-ml beaker. Thirty ml of carbon tetrachloride shall be added. The beaker and contents shall be placed on a steam bath and allowed to heat gently while covered with a watch glass. After 5 minutes the beaker and contents shall be removed and allowed to cool to room temperature. The carbon tetrachloride solution shall be filtered through a tared medium porosity crucible. The beaker shall be washed carefully with a stream of carbon tetrachloride making certain that all of the residue in the beaker is transferred to the crucible. The crucible and contents shall be washed with two additional 20 ml portions of carbon tetrachloride. The carbon tetrachloride shall be allowed to come in contact with the sample for about one minute before applying suction. The crucible shall be aspirated until the odor of carbon tetrachloride is no longer detectable. The crucible and contents shall be dried in an oven maintained at  $100^{\circ} \pm 5^{\circ}\text{C}$  for one hour, cooled in a desiccator and weighed. The RDX content shall be calculated as follows:

$$\text{Percentage of RDX} = \frac{(A-B)}{W} 100$$

where:

- A = weight of the crucible and residue  
 B = weight of the crucible empty  
 W = weight of sample on a moisture-free basis

4.5.2 Engine oil content.-The oil content shall be determined by difference by adding the percentages of RDX and moisture and subtracting the sum from 100 percent.



4.6 Acidity.—An accurately weighed portion of approximately 10 grams of the sample shall be transferred to an 800-ml beaker. Two hundred fifty ml of acetone shall be added to the beaker. The beaker shall be covered with a watch glass and heated in a steam bath until the contents are completely dissolved. The sample shall be stirred occasionally. One hundred ml of carbon tetrachloride shall be added, stirred to insure homogeneity and then 400 ml of water shall be added. The first 200 ml of water should be added slowly with vigorous stirring to insure complete washing of the carbon tetrachloride. The final 200 ml may be added more rapidly. The beaker and contents shall be cooled to room temperature. A mixed indicator shall be prepared by dissolving 0.15 gram of powdered cresol green and 0.10 gram of powdered methyl red in a small amount of 0.01N sodium hydroxide solution. The indicator solution shall be neutralized to the blue green end point, and made up to 250 ml with water. Twelve drops of mixed indicator shall be added to the water layer in the 800 ml beaker and titrated with 0.05 normal sodium hydroxide to a blue green end point. Concurrently, a blank determination shall be run for correction of any acidity in the solutions. The acidity shall be calculated as percent acetic acid as follows:

$$\text{Acidity (as percent acetic acid)} = \frac{6.0 (V-v)N}{W}$$

where:

- V = ml sodium hydroxide used in sample titration
- v = ml of sodium hydroxide used in the blank titration
- N = normality of the sodium hydroxide solution
- W = weight of the sample

4.7 Inorganic insoluble matter.—An accurately weighed portion of approximately 10 grams of the sample shall be transferred to a 600-ml beaker. Two hundred fifty ml of acetone and 100 ml of carbon tetrachloride shall be added. The beaker shall be covered with a watch glass, placed on a steam bath and stirred occasionally until all of the sample appears to be in solution. The solution shall be filtered through a tared porcelain filtering crucible which has been previously ignited to 600°C. All insoluble material shall be transferred to the crucible. The crucible shall be washed with a mixture of warm acetone-carbon tetrachloride. The crucible shall be aspirated until the odor of acetone and carbon tetrachloride are not detectable. The crucible and residue shall be placed in a muffle furnace at 600°C for one hour, cooled to room temperature and weighed. The increase in weight of the crucible shall be calculated to percent inorganic insoluble matter in the sample.

## 5. PREPARATION FOR DELIVERY

5.1 Packing.—Desensitized RDX in bulk form for shipment shall be packed and marked in accordance with Ordnance Corps Drawings 76-1-1405, 76-1-1406, or 76-1-1407, as specified (see 6.1), in wooden boxes or fiber cartons of 50 pounds net capacity, and so constructed as to comply in all respects with the Interstate Commerce Commission Regulations, as specified in the Code of Federal Regulations 49 CFR 71-78. No container shall contain desensitized RDX from more than two batches.

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5.2 Labeling and marking.- In addition to any special marking required by the contract or purchase order, shipments shall be marked in accordance with ICC requirements, as stipulated in the Code of Federal Regulations 49 CFR 71-78, and with Standard MIL-STD-129. Each container shall be clearly marked and labeled to indicate the batch and lot number.

## 6. NOTES

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

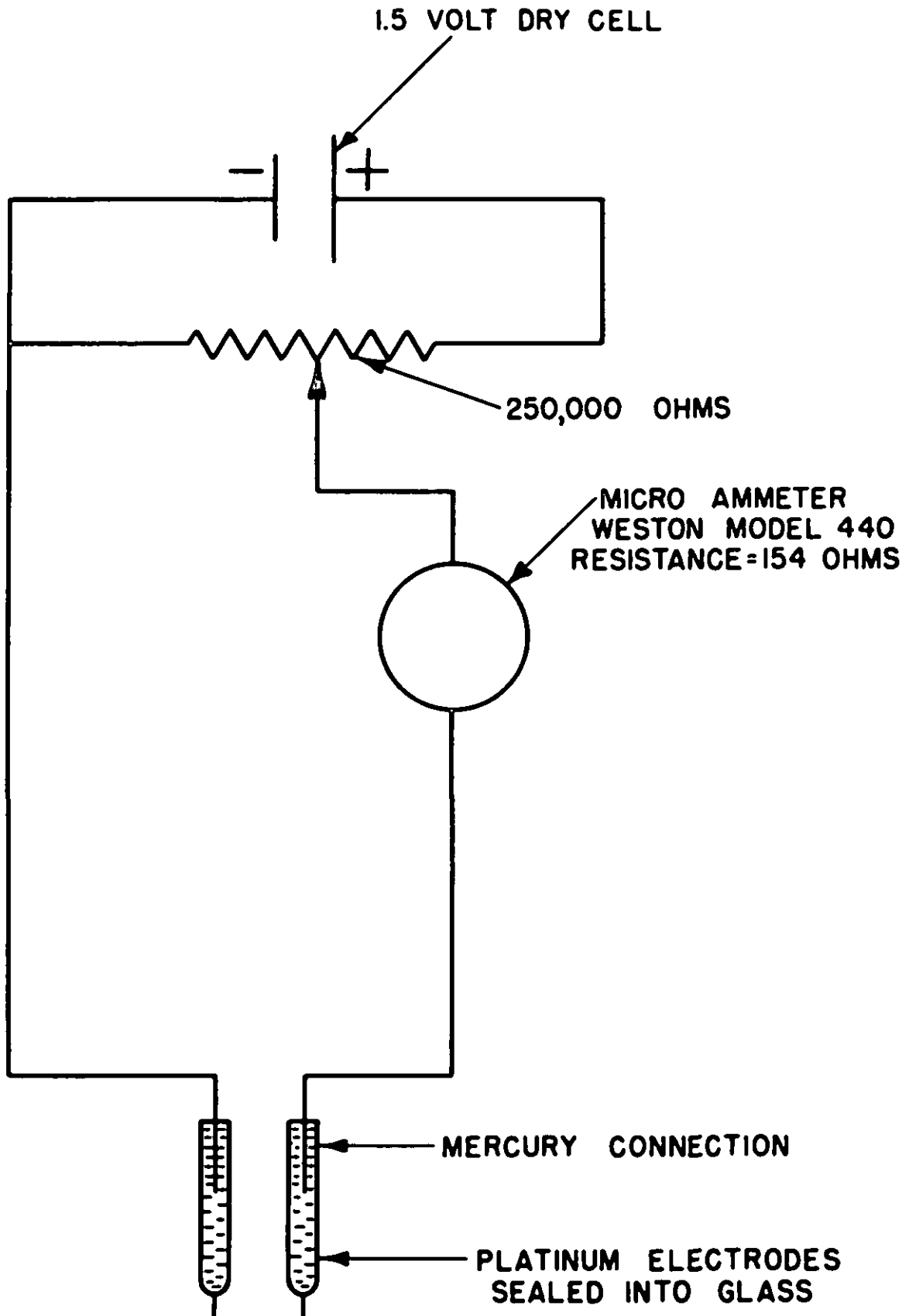
- a. Title, number, and date of this specification.
- b. Packing required (see 5.1).

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 in any way be related thereto.

Custodian:

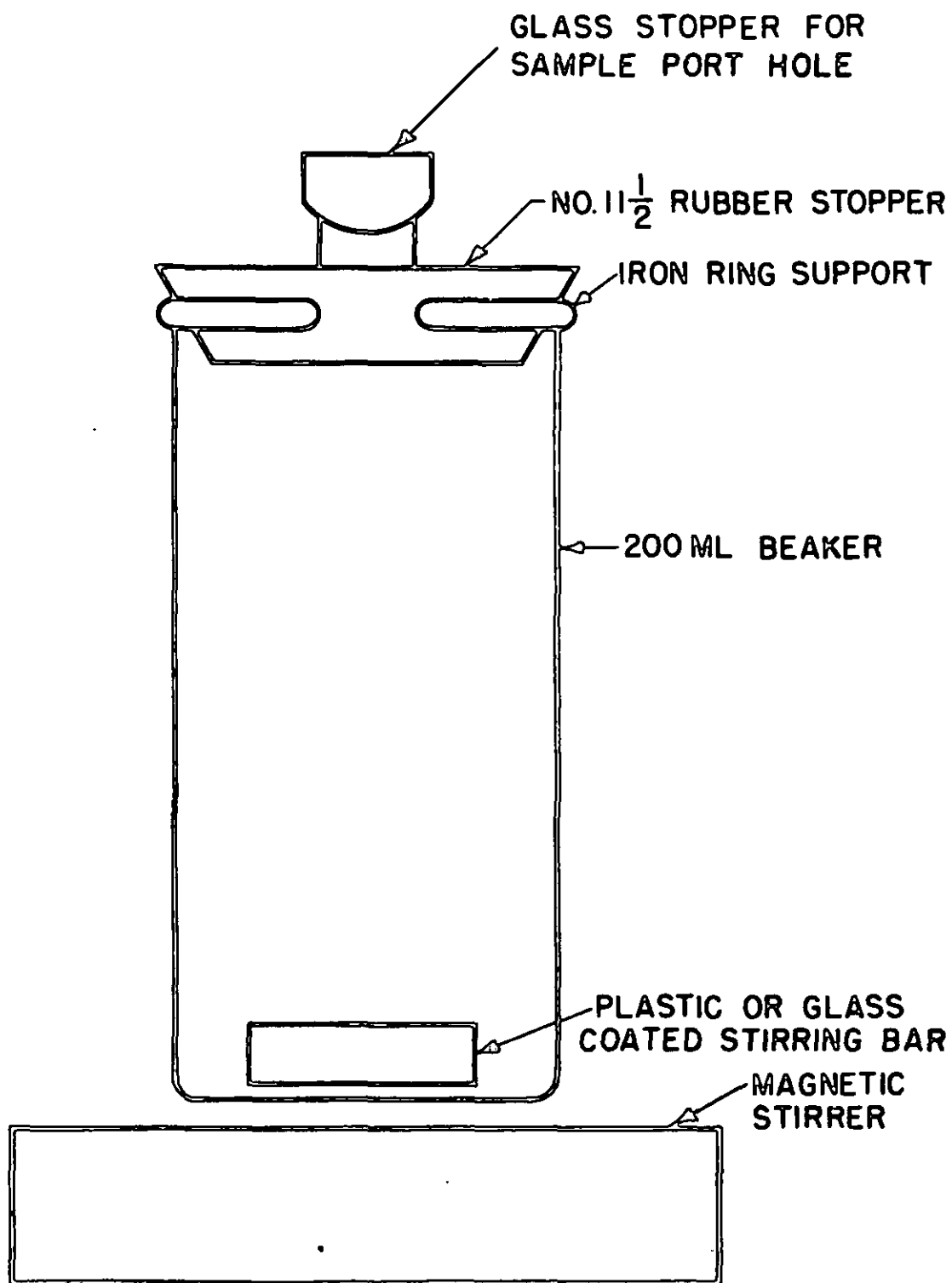
Army-Ordnance Corps





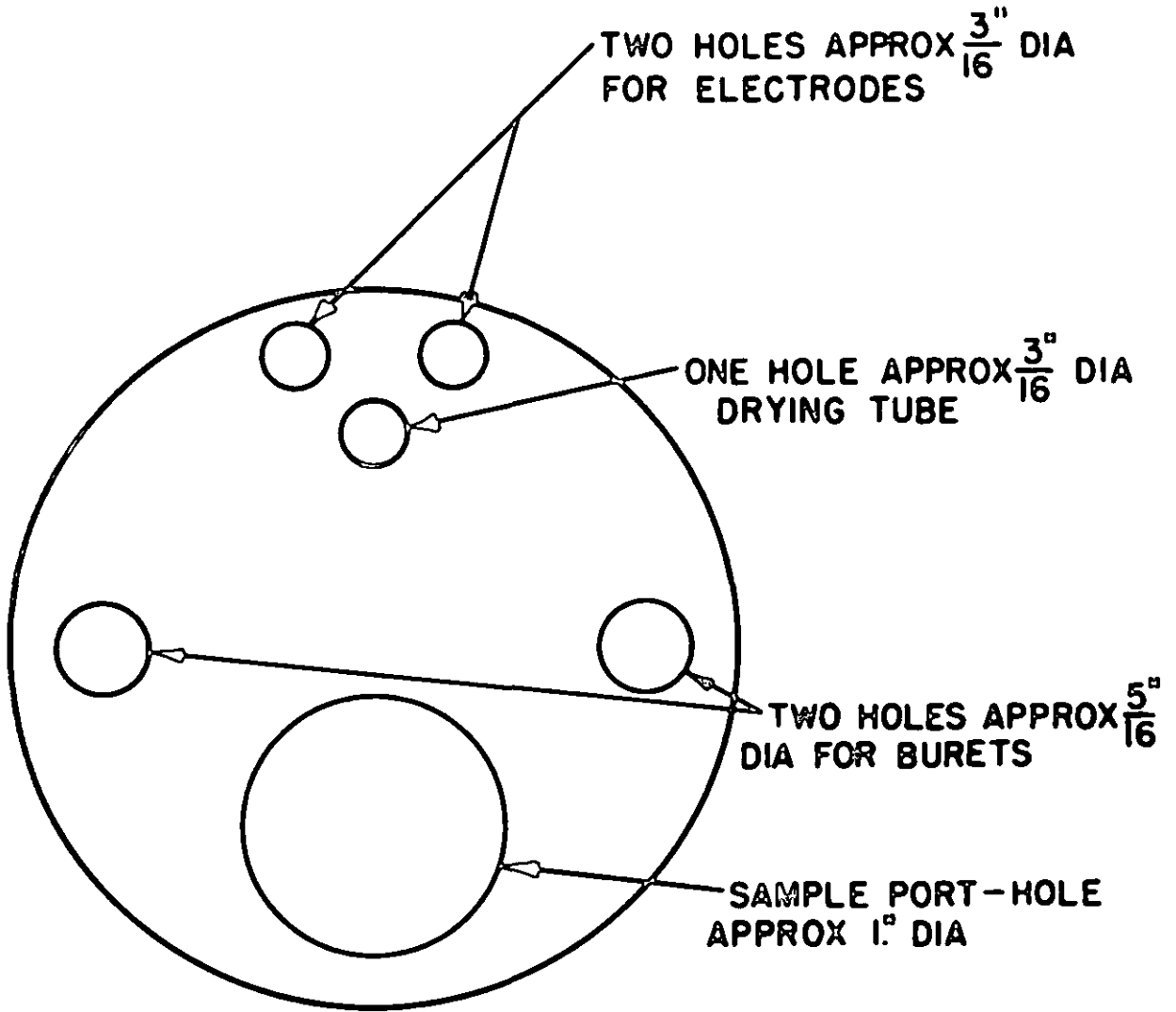
WIRING  
FIGURE 1

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TITRATION ASSEMBLY

FIGURE 2



RUBBER STOPPER-TOP VIEW  
FIGURE 3