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AUTHORITY

USNWC ltr, 30 Aug 1974

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SPECIFICATION

1 of 1

1. COMPONENT/PART NAME PER GENERIC CODE: Propulsion Parts & Materials, Solid Fuel Engines Propellants	2. PROGRAM OR WEAPON SYSTEM: Multiple	D0719			
4. ORIGINATOR'S SPECIFICATION TITLE: Purchase Description - 2-(2,4-Dinitrophenoxy) Ethanol	5. ORIGINATOR'S SPEC. NO.: WS 7653	3. DATE OF:	DAY	MO.	YR.
		ISSUE	13	10	67
		REVISION			

SPECIFICATION IS: ☐ DRAFT ☐ PRELIMINARY ☒ FINAL

7. THIS SPECIFICATION COMPLEMENTS REPORT NO:

8. TYPE OF SPECIFICATION

- ☒ (A) GENERAL PRODUCT REQUIREMENTS FOR A FAMILY OF PARTS - PROCUREMENT DOCUMENT
- ☐ (B) INDIVIDUAL DETAIL PARTS DOCUMENT; STDS BOOK PAGES - FOR PROCUREMENT
- ☐ (C) DETAIL INSPECTION, PROCESS CONTROL, AND/OR TEST PROCEDURES FOR SPECIFIC PARTS
- ☐ (D) PROCESS (PAINTING, WELDING, FINISHING, HEAT TREATING ETC.) APPLICABLE TO MANY PARTS
- ☐ (E) SPEC. FOR PERFORMANCE, RELIABILITY, AND/OR ENVIRONMENT FOR ASSEMBLIES, EQUIPMENTS, SUBSYSTEMS AND SYSTEMS
- ☐ (F) PERFORMANCE AND APPLICATION DATA FOR DESIGN ENG. USE ON PARTS - NOT FOR PROCUREMENT
- ☐ (G) OTHER (DETAIL IN 10.)

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1961 Book of ASTM Std. Part 27			x	
1964 Book of ASTM Std. Part 17			x	

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Naval Fleet Missile Comd.
Analysis and Evaluation Group
Corona, Calif 91720
FDEP Office

11. SIGNED: R. S. Harper 11/6/68

12. CONTRACTOR: NWC/CL | SUBCONTRACTOR:

13. SPEC. NO. 565.70.00.00-X7-05

NOTICES PAGE

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Code Ident 30003 WS 7653

NAVAL AIR SYSTEMS COMMAND

DEPARTMENT OF THE NAVY

PURCHASE DESCRIPTION

2-(2,4-DINITROPHENOXY) ETHANOL

1. SCOPE.

1.1 Scope. This purchase description covers one grade of 2-(2,4-dinitrophenoxy) ethanol.

2. APPLICABLE DOCUMENTS.

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

STANDARDS

Military

MIL-STD-129 Marking for Shipment and Storage.

(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.)

FSC 6810

2.2 Other publications. The following documents form a part of this document to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials (ASTM)

1961 Book of ASTM Standards; Part 27	Test-Method ASTM D1303-55, "Total Chlorine in Vinyl Chloride Polymers and Copolymers".
1964 Book of ASTM Standards; Part 17	Test-Method ASTM D128-64, "Analysis of Lubricating Grease".

(ASTM Publications are published by the American Society for Testing and Materials, Philadelphia 3, Pennsylvania.)

3. REQUIREMENTS.

3.1 Preproduction sample. Unless otherwise specified (see 6.2), a preproduction sample shall meet all requirements of this document. The preproduction sample shall be prepared using the same methods and procedures proposed for production. Any production prior to acceptance of the preproduction sample shall be at the risk of the supplier.

3.2 Data. No data is required by this document or by referenced documents in section 2 unless specified in the contract or purchase order.

3.3 Compliance to documents. 2-(2,4-dinitrophenoxy) ethanol shall conform to the requirements herein and to the applicable requirements of documents listed in section 2.

3.4 Product characteristics and performance. When tested in accordance with 4.7 of this document 2-(2,4-dinitrophenoxy) ethanol shall meet the following product characteristics and performance.

3.4.1 Chemical and physical analysis. The chemical and physical analysis of the material shall be specified in Table I.

4

Table I. Chemical and Physical Analysis

Characteristic, %	Minimum	Maximum
Chlorine		0.50
Ash		0.20
Moisture		0.10
Reagent acetone insolubles		0.20
1,2-bis(2,4-dinitrophenoxy)ethane	*0	40

*The 0 to 40 percent range is the maximum range for 1,2-bis(2,4-dinitrophenoxy)ethane. The specific range or percent 1,2-bis(2,4-dinitrophenoxy)ethane shall be as specified in the contract or purchase order (see 6.2).

3.5 Workmanship. The 2-(2,4-dinitrophenoxy) ethanol shall be uniform in quality, free from foreign materials, and shall be manufactured under conditions and procedures standard in the industry.

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, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in this document where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Lot. A lot shall consist of material produced at one plant with no change in formulation or process. If manufacture is by batch process, each batch shall constitute a lot. A batch shall be as defined in 6.3.

4.3 Acceptance sampling. The number of containers to be chosen at random for acceptance sampling shall be equal to the square root of the total number of containers in the lot. If the number thus obtained is not a whole number, the number of containers to be sampled shall be increased to the next higher whole number. In no case, however, shall the number of containers to be sampled be less than seven (unless there are less than seven containers in the lot, in which case, each container shall be sampled).

4.3.1 Primary sample. From each selected container, a sample shall be taken from three or more places throughout the container. The total weight of the samples taken from each container shall weigh at least 50 grams (gm). Each sample thus taken shall be mixed thoroughly, placed in a clean dry container, and labeled to identify the material name, original container designation, contract number, and lot number.

4.3.2 Composite sample. Each primary sample shall be subdivided to prepare a composite sample (not in excess of 350 gm). Primary material not used shall be returned to the primary sample container. After mixing the composite sample thoroughly, the composite sample shall be placed in a clean, dry container and sealed. The composite sample shall be identified with the material name, container designation, contract number, and lot number. All specified chemical tests shall be made of this composite sample representing the lot. Failure of the composite sample to pass all of the tests herein shall result in rejection of the lot represented.

4.4 Classification of tests. Inspection and testing of 2-(2,4-dinitrophenoxy) ethanol shall be classified as follows:

(a) Preproduction tests.

(b) Quality conformance tests.

4.5 Preproduction tests. Preproduction tests shall be conducted only on the preproduction sample and shall consist of all examinations and tests specified in 4.6.

6

4.6 Quality conformance tests. Quality conformance tests for acceptance of the 2-(2,4-dinitrophenoxy) ethanol shall consist of the following tests:

Characteristic	Test
Chlorine	4.7.1
Ash	4.7.2
Moisture	4.7.3
Reagent acetone insolubles	4.7.4
1,2-bis(2,4-dinitrophenoxy)ethane	4.7.5

4.7 Tests. The following procedures shall be used to determine that the requirements of this document have been met. Any proposed change in test procedures or equipment shall necessitate, before adoption, prior approval of the procuring activity. In case of dispute between the results from any proposed method or equipment and what is cited herein, the results using the methods and the equipment specified in this document shall prevail. Unless otherwise specified, all tests shall be run in duplicate. The average of the two results shall be taken as the test result.

4.7.1 Chlorine. Determine the amount of chlorine in accordance with the procedure given in Test Method D1303-55 "Total Chlorine in Vinyl Chloride Polymers and Copolymers" (Part 27 of ASTM, page 472), except use ferric alum indicator in place of ferric nitrate indicator, and electrical ignition of the Parr bomb in place of ignition by use of a Bunsen burner.

4.7.1.1 Acceptance criteria. For the lot represented to pass the chlorine test, the value obtained for the percent chlorine shall be no greater than the value specified in 3.4.1.

4.7.2 Ash. Determine the amount of ash in accordance with the procedure given in Test Method D128-64 "Analysis of Lubricating Grease" (Part 17 of ASTM, page 54).

4.7.2.1 Acceptance criteria. For the lot represented to pass the ash test, the value obtained for percent ash shall be no greater than the value specified in 3.4.1.

7

4.7.3 Moisture.

4.7.3.1 Reagents.

(a) Toluene, dried to contain less than 0.005 percent water.

(b) "Desicote", Beckman Instruments, Inc., Fullerton, California.

(c) Alcoholic potassium hydroxide (KOH) solution. Place 240 gm of a 50 percent distilled water solution of KOH in a 1000-milliliter (ml) volumetric flask. Dilute to exact volume with 95 percent ethyl alcohol.

4.7.3.2 Apparatus.

(a) Round-bottom flask (500-ml) with 24/40 standard-taper-ground-glass joint (with stopper).

(b) Heating mantle, hemispherical, 110-volt, 250-watt (for 500-ml round-bottom flask), Glascol brand or equivalent.

(c) Variable auto-transformer, 110-volt, 7.5-ampere.

(d) Condenser, West type, 24/40 standard-taper-ground-glass joints with a jacket approximately 300 millimeters (mm) in length, Corning No. 2740 or equivalent. (See Figure 1.)

(e) Moisture trap. (See Figure 2.)

4.7.3.3 Special instructions. To the moisture-determination apparatus, apply "Desicote" (4.7.3.1 (b)) in the following manner to render it water repellant:

(a) To clean the apparatus before coating, put clean cork stoppers in the outlet of the condenser (4.7.3.2 (d) and Figure 1) and the vapor-inlet sidearm of the trap (4.7.3.2 (e) and Figure 2). Completely fill both the condenser and the trap of the disassembled apparatus with alcoholic-KOH solution (4.7.3.1 (c)).

When preparing the moisture trap (after it is filled with solution) quickly insert a clean cork stopper into the trap at point C to push solution through the capillary tube and to cause the trap to remain full of solution. Allow the solution to stay in place in both pieces of apparatus approximately 15 minutes. Drain the solution, rinse the pieces well with cold tap water, and rinse twice with distilled water. Dry the pieces at approximately 100 degrees centigrade (°C) (212 degrees Fahrenheit (°F)), cool to room temperature and draw dry air through them for approximately 15 minutes.

(b) In order to coat the cleaned condenser with "Desicote", put a clean cork stopper into one end, fill with "Desicote" (4.7.3.1 (b)), and allow to stand for not less than 10 minutes. Drain, returning the used "Desicote" to the container for reuse. (Avoid any unnecessary exposure of "Desicote" to the atmosphere.) Remove the stopper, then allow the condenser to stand in a vertical position for approximately 2 hours at room temperature; then dry at approximately 100°C (212°F) for several hours.

(c) To coat the cleaned moisture trap, place the clean cork stopper into the end of the vapor-inlet sidearm (point A, Figure 2), hold the trap in a vertical position, and fill it completely with "Desicote". As was done above with the alcoholic-KOH solution, quickly insert a clean stopper (at point C, Figure 2) to force through the capillary tube and to cause the trap to remain full of solution. Allow the trap to stand for at least 10 minutes. Return the used "Desicote" to its container. Remove stoppers, then let the trap dry in a vertical position at room temperature for approximately 2 hours. At approximately 15-minute intervals, remove any solution that drains to the bottom of the capillary tube. Then dry at approximately 100°C (212°F) for several hours.

4.7.3.4 Procedure. Determine water content by the Dean-Stark method of analysis. Add approximately 100 gm of granular sample (weighed to nearest 0.01 gm) to 150 gm of dry toluene (4.7.3.1 (a)) in a 500-ml round-bottom flask (4.7.3.2 (a)). Add a few carborundum chips to aid smooth boiling. Stopper the flask and mix the contents thoroughly. Place the flask in a heating mantle (4.7.3.2 (b)) which is controlled by a variable transformer (4.7.3.2 (c)). Attach a piece of rubber tubing approximately 4 inches long to the tip of the calibrated capillary tube of the moisture trap (4.7.3.2 (e)). Close the tubing with a pinch clamp. To the specially prepared moisture trap, add approximately 2 ml of dry toluene (4.7.3.1 (a)). With a small piece of wire, place a very minute amount of finely-ground methylene blue in the bottom of the moisture trap. Assemble the apparatus as shown in Figure 1, provide a flow of water through the specially prepared condenser (4.7.3.2 (d)), and adjust the variable transformer to provide approximately 70 volts. Reflux the sample with the toluene for 3 hours at a rate of 120 drops of toluene per minute. After removing the desiccant tube, wash the condenser walls with three 5- to 10-ml portions of dry toluene. Using the fingers, compress the piece of rubber tubing above the clamp to force the water column back and forth in the bottom of the moisture trap until all the water has collected and the methylene blue has become thoroughly mixed with the water. Then slowly release the pinch clamp until the blue-tinted water (as an unbroken column) moves into the graduated capillary. Read and record the volume of water to nearest 0.01 ml.

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

Where: V = Volume of water collected, ml

W = Weight of sample taken, gm

4.7.3.5 Acceptance criteria. For the lot represented to pass the moisture test, the value obtained for the percent moisture shall be no greater than the value specified in 3.4.1.

4.7.4 Reagent acetone insolubles. Weigh 5 gm of dry sample (weighed to the nearest 0.0001 gm) into a 250-ml Erlenmeyer flask. Add 100 ml of reagent grade acetone. Boil the mixture gently for 15 minutes on a hot plate. Filter the hot mixture rapidly through a tared fritted glass filter using strong suction and a 500-ml suction flask. Rinse out any remaining residue in the Erlenmeyer flask with 20 to 50 ml of hot acetone and filter the washings in the same manner as before. Place the funnel and residue in an oven at 110°C (230°F) and dry for 20 minutes. Carefully reweigh the funnel and residue to the nearest 0.0001 gm.

$$\text{Percent reagent acetone insoluble} = \frac{(A-B)\ 100}{C}$$

Where: A = Weight of funnel plus residue, gm

B = Weight of funnel, gm

C = Weight of sample, gm

4.7.4.1 Acceptance criteria. For the lot represented to pass the reagent acetone insolubles test, the value obtained for the percent reagent acetone insolubles shall be no greater than the value specified in 3.4.1.

4.7.5 1,2-bis(2,4-dinitrophenoxy)ethane. The percentage of 1,2-bis(2,4-dinitrophenoxy)ethane specified in 3.4.1 shall be determined in accordance with 4.7.5.1.

4.7.5.1 Procedure. Weigh approximately 10 gm of dry sample (weighed to the nearest 0.1 gm) into a 250-ml Erlenmeyer flask. Add 100 ml of toluene which has been dried over sodium wire. Boil the mixture gently for 15 minutes on a hot plate. Filter the hot mixture rapidly through a Buchner funnel or fritted glass filter using strong suction and a 500-ml suction flask. Rinse out any remaining residue in the Erlenmeyer flask with 20 to 50 ml of hot, dry toluene and filter the washings in the same manner as before. Place the funnel and residue in an oven at 110°C (230°F) and dry for 30 minutes. Carefully scrape out the residue into a tared container and reweigh.

$$\text{Percent 1,2-(2,4-dinitrophenoxy)ethane} = \frac{A\ (100)}{B}$$

Where: A = Weight of residue, gm

B = Weight of sample, gm

4.7.5.2 Acceptance criteria. For the lot represented to pass the 1,2-(2,4-dinitrophenoxy)ethane test, the value obtained for the percent 1,2-(2,4-dinitrophenoxy)ethane shall be within the range specified in the contract or purchase order (see 6.2).

4.8 Packing and marking. Determine that all packing and marking conforms to section 5 of this document.

5. PREPARATION FOR DELIVERY.

5.1 Preservation and packaging. Not applicable (unless specified in the contract or purchase order).

5.2 Packing.

5.2.1 Level A. Not applicable.

5.2.2 Level B. Not applicable.

5.2.3 Level C. The material shall be packed as directed in the contract to afford protection against damage during direct shipment from the supply source to the first receiving activity for immediate use. Containers shall comply with common carrier regulations applicable to the mode of transportation to be used. (See 6.2.)

5.3 Marking. In addition to the markings required by contract or purchase order, unit packages and shipping containers shall be marked in accordance with the requirements of MIL-STD-129.

6. NOTES.

6.1 Intended use. 2-(2,4-dinitrophenoxy) ethanol is intended for use as a plasticizer in ammonium-nitrate-based solid propellants.

6.2 Ordering data. Procurement documents should specify the following:

(a) Title, number and date of this document.

(b) Whether a preproduction sample is required (see 3.1).

(c) The range of 1,2-bis(2,4-dinitrophenoxy)ethane content (see 3.4.1).

(d) Type and size of shipping container (see 5.2.2).

(e) Invocation of MIL-A-9858.

6.3 Definitions

6.3.1 Batch. A batch is defined as that quantity of material which has been subjected to one or more chemical or physical processes (or combinations thereof) intended to produce a desired product having substantially uniform characteristics. The final step in the processing must have treated the entire contents of the batch at one time.

6.4 Safety and health warning. Handling of the chemicals specified herein shall be in accordance with suitable safety and health precautions.

6.5 Acceptance product. An acceptable product under this document is 2-(2,4 dinitrophenoxy) ethanol manufactured by Amoco Chemicals Corporation, Seymour, Indiana.

Custodian:
NASC 52021E

Preparing Activity:
NWC/China Lake, California

B

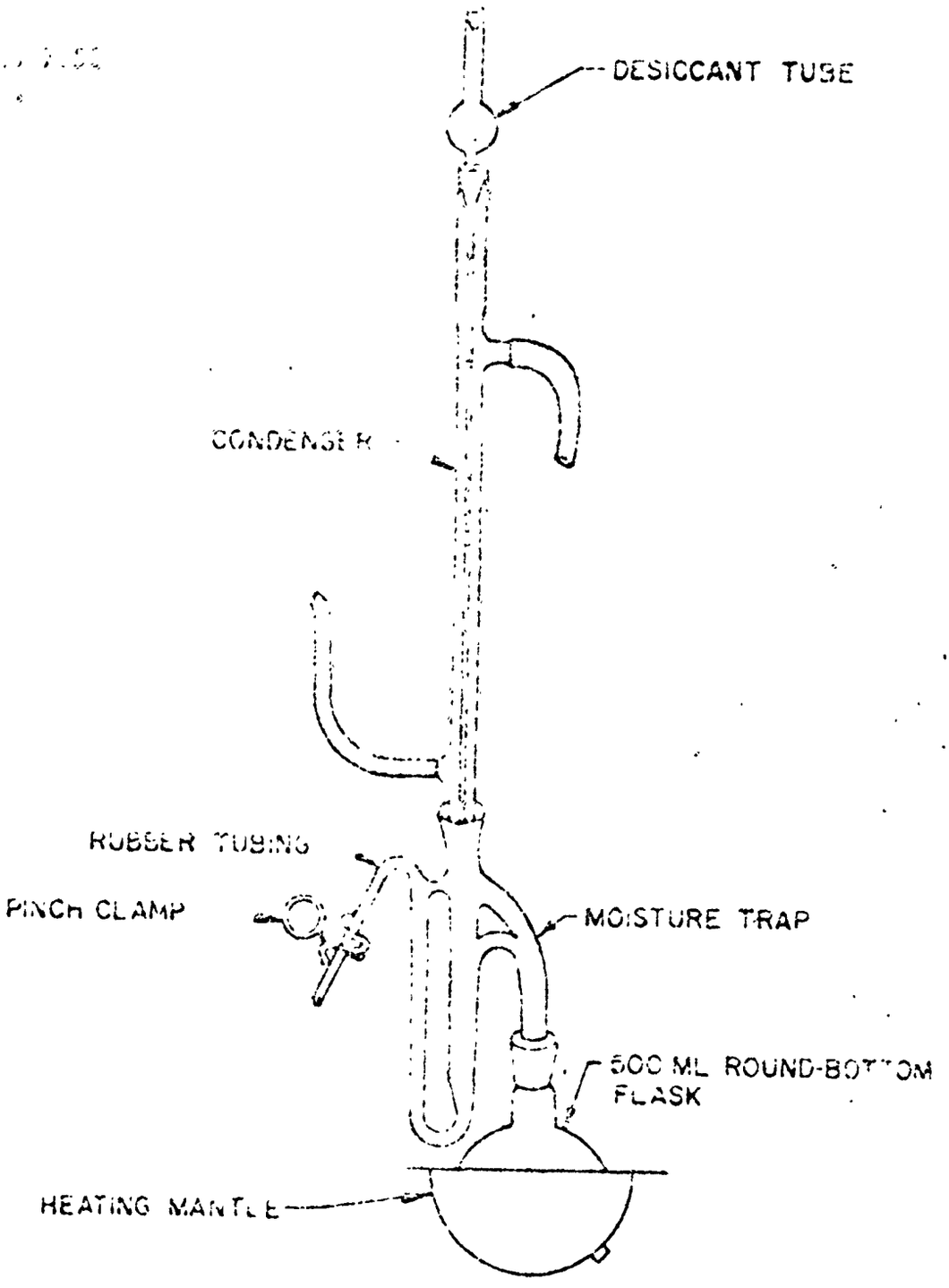

FIGURE 1
MOISTURE DETERMINATION APPARATUS

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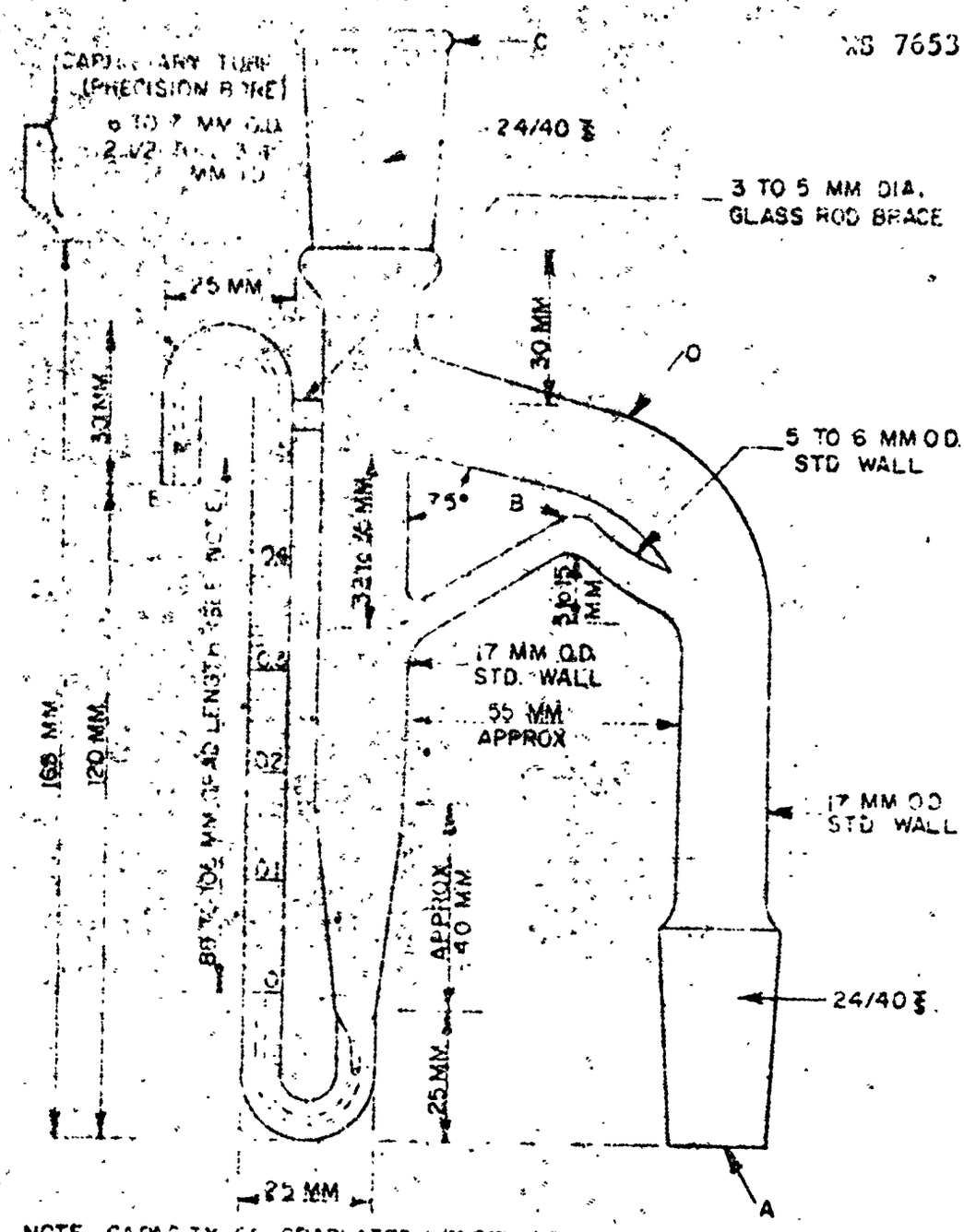

NOTE: CAPACITY OF GRADUATED LENGTH OF CAPILLIARY TUBE = 0.5 ML. PRINCIPAL GRADUATIONS IN TENTHS (SUBDIVIDED INTO HUNDREDTHS)

FIGURE 2. MOISTURE TRAP