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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	D0720			
	5. ORIGINATOR'S SPEC. NO.: WS 7652	3. DATE OF: ISSUE	DAY 13	MO 10	YR 67
4. ORIGINATOR'S SPECIFICATION TITLE: Purchase Description - Sodium Barbiturate		REVISION			
	6. 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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11. SIGNED	12. CONTRACTOR	SUBCONTRACTOR
R. S. Harper 11/8/68	NWC/CL	

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Code Ident 30003 WS 7652

NAVAL AIR SYSTEMS COMMAND

DEPARTMENT OF THE NAVY

PURCHASE DESCRIPTION

SODIUM BARBITURATE

1. SCOPE.

1.1 Scope. This purchase description covers one grade of granular sodium barbiturate.

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

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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. Sodium barbiturate 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, sodium barbiturate shall meet the following product characteristics and performance.

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

Table I. Chemical Analysis

Characteristic	Minimum	Maximum
Sodium barbiturate, %	96.6	
Metals, (as sodium) %	14.8	15.8
Moisture, %	--	0.5
pH number	8.0	10.0

3.5 Workmanship. The sodium barbiturate shall be uniform in quality, free from foreign materials, and shall be manufactured under conditions and procedures standard in the industry.

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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 500 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 on 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 sodium barbiturate shall be classified as follows:

(a) Preproduction tests.

(b) Quality conformance tests.

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

4.6 Quality conformance tests. Quality conformance tests for acceptance of the sodium barbiturate shall consist of the following tests:

Characteristics	Test
Sodium barbiturate	4.7.1
Sodium	4.7.2
Moisture	4.7.3
pH	4.7.4

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.

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4.7.1 Sodium barbiturate.

4.7.1.1 Equipment.

(a) Steam bath.

(b) Tared weighing bottle.

(c) Analytical balance.

(d) Watch glass - 75-millimeter (mm) diameter.

(e) Burette - 5-milliliter (ml), graduated to 0.01 ml (S.G.H.C. No. B7860).

(f) Magnetic stirrer.

(g) Stirring bars - teflon covered.

(h) Beaker - 150-ml.

(i) Microscope lamp - A.O. Spencer No. 353, or equivalent, with yellow filter.

4.7.1.2 Reagents.

(a) Standard N/10 perchloric acid ($HClO_4$) in acetic acid.

(b) Crystal violet -o- nitroaniline indicator.

(c) Acetic anhydride - reagent grade.

(d) Toluene - reagent grade.

(e) Acetic acid - reagent grade.

(f) Sample.

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4.7.1.3 Procedures.

(a) Weigh (to the nearest 0.0001 gm) 0.0250 to 0.0300 gm of sample in a tared, dry, weighing bottle on the analytical balance. Transfer the sample to a 150-ml beaker and reweigh the bottle. The difference in weight is the weight of sample. Add 10 ml of acetic acid and 1 ml of acetic anhydride to the beaker and warm on the steam bath until the sample is completely dissolved.

(b) Add 50 ml of toluene and continue warming on the steam bath for about 10 minutes. Cover the beaker with a watch glass.

(c) Transfer the beaker to the magnetic stirrer and focus the light through the beaker to an intense spot on the front of the beaker. Fasten a piece of white paper around the beaker to aid in focusing the light. Add 3 drops of crystal violet o-nitroaniline indicator and titrate dropwise with N/10 $HClO_4$ to the green end point. The end point is a definite yellow green - not blue green.

$$\text{Percent sodium barbiturate} = \frac{A \times 1.5}{B}$$

Where: A = Volume of N/10 $HClO_4$ used in titration, ml.

B = Weight of sample, gm.

4.7.1.4 Acceptance criteria. For the lot represented to pass the sodium barbiturate test, the value obtained for percent sodium barbiturate shall be not less than the value specified in 3.4.1.

4.7.2 Metals (as sodium).

(a) Place approximately 0.5 gm of sample (weighed to the nearest 0.0001 gm) into a clean, dry, tared, Vycor crucible.

(b) Add approximately 1.0 ml of reagent-grade concentrated sulfuric acid to the sample and place the crucible on a hot plate turned to high heat.

(c) Allow the crucible to remain on the hot plate until bubbling is no longer apparent (approximately 2 to 4 hours).

(d) Transfer the crucible to a muffle furnace set at 500 to 600 degrees centigrade (°C) (932 to 1112 degrees Fahrenheit (°F)). Leave the crucible in the furnace until any existing black ash has disappeared and only a white ash remains.

(e) Increase the temperature of the muffle furnace to 800 to 900°C (1472 to 1652°F). Leave the crucible in the furnace at this temperature for an additional one-half hour or until the white ash has fused together.

(f) Transfer the crucible to a desiccator, cool, then reweigh to the nearest 0.0001 gm.

$$\text{Percent metals (as sodium)} = \frac{32.38\ (A-B)}{C}$$

Where: A = Weight of crucible plus residue, gm.

B = Weight of crucible empty, gm.

C = Weight of sample taken, gm.

4.7.2.1 Acceptance criteria. For the lot represented to pass the metals (as sodium) test, the value obtained for the percent metal (as sodium) shall be within the range shown in 3.4.1.

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 solution of KOH in distilled water into a 1000-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 repellent:

(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 (see Figure 2) 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°C (212°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 side-arm 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 "Desicote" 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.

NOTE. Should the coated surface become dirty, its water repellency is impaired. It may be cleaned and restored to usefulness with a non-alkaline detergent. Gentle brushing may also be used, if necessary, but a thorough rinsing with the non-alkaline detergent solution should be sufficient.

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.

NOTE. The apparatus must be rendered water repellent before use. For special instructions for rendering the glass condenser and moisture trap water repellent, see 4.7.3.3.

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 together (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 the record the volume of water to nearest 0.01 ml.

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

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 percent moisture shall be no greater than the value specified in 3.4.1.

4.7.4 pH determination. Dissolve approximately one gm of the granular sodium barbiturate (weighed to the nearest 0.01 gm) in 100 ml of freshly boiled distilled water contained in a 200-ml beaker. Determine the pH of the solution electrometically using a pH meter.

4.7.4.1 Apparatus. Electronic pH apparatus, Beckman Zeromatic, Beckman Instruments, Inc., Fullerton, California, or approved equivalent.

4.7.4.2 Acceptance criteria. For the lot represented to pass the pH determination test, the value obtained for the pH number shall be within the range shown in 3.4.1.

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. This sodium barbiturate is intended for use as a catalyst 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 preproduction tests are required (see 3.1).

(c) Type and size of shipping containers (see 5.2.3).

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 combination 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 Approved product. Approved products under this document are sodium barbiturate, acid free, manufactured by the Kay Fries Chemical Company, New York, New York and sodium barbiturate, acid free, manufactured by the Abbott Laboratories, North Chicago, Illinois.

Custodian:
NASC 52021E

Preparing Activity:
NWC/China Lake, California

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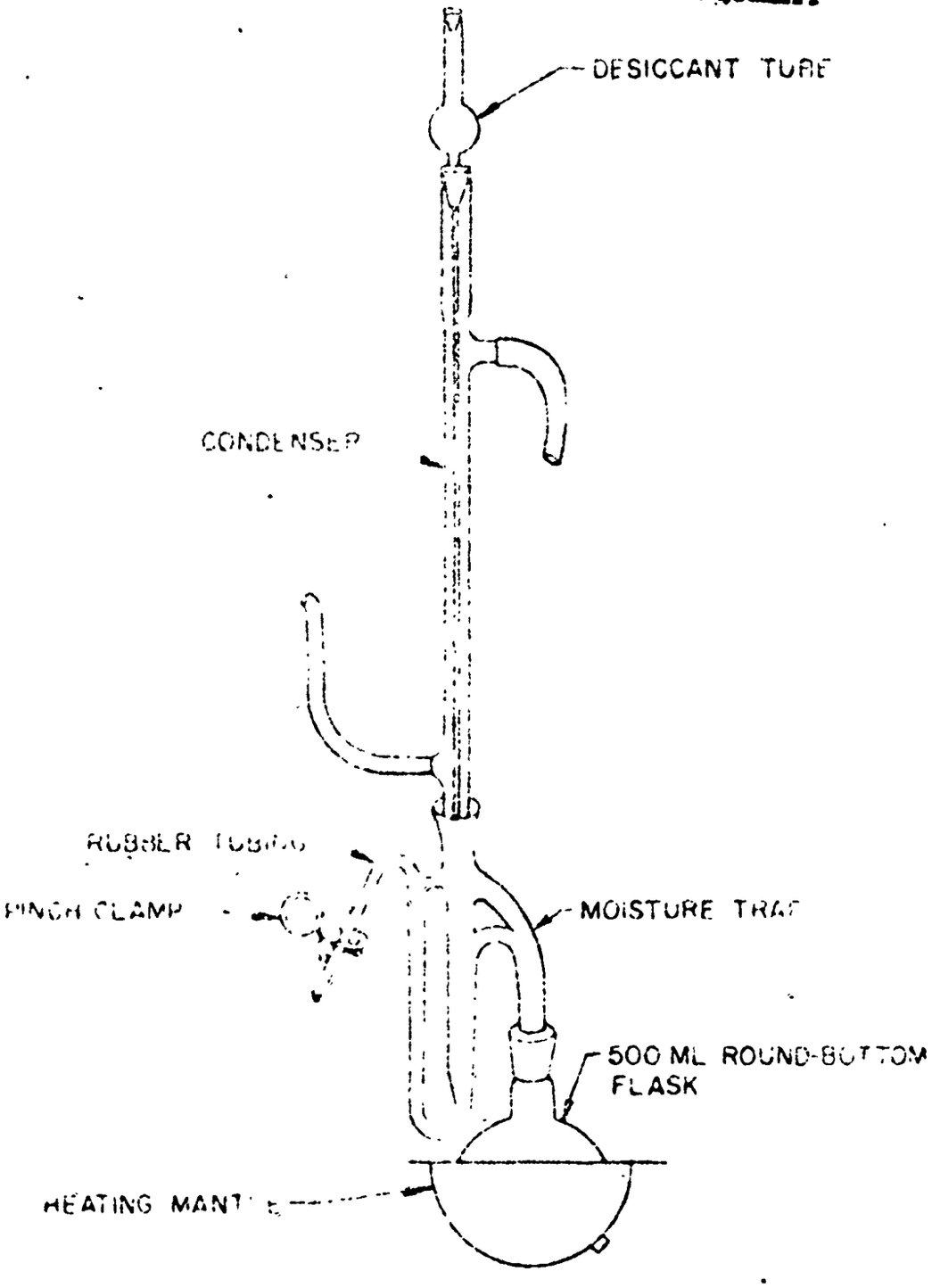

FIGURE 1
MOISTURE DETERMINATION APPARATUS

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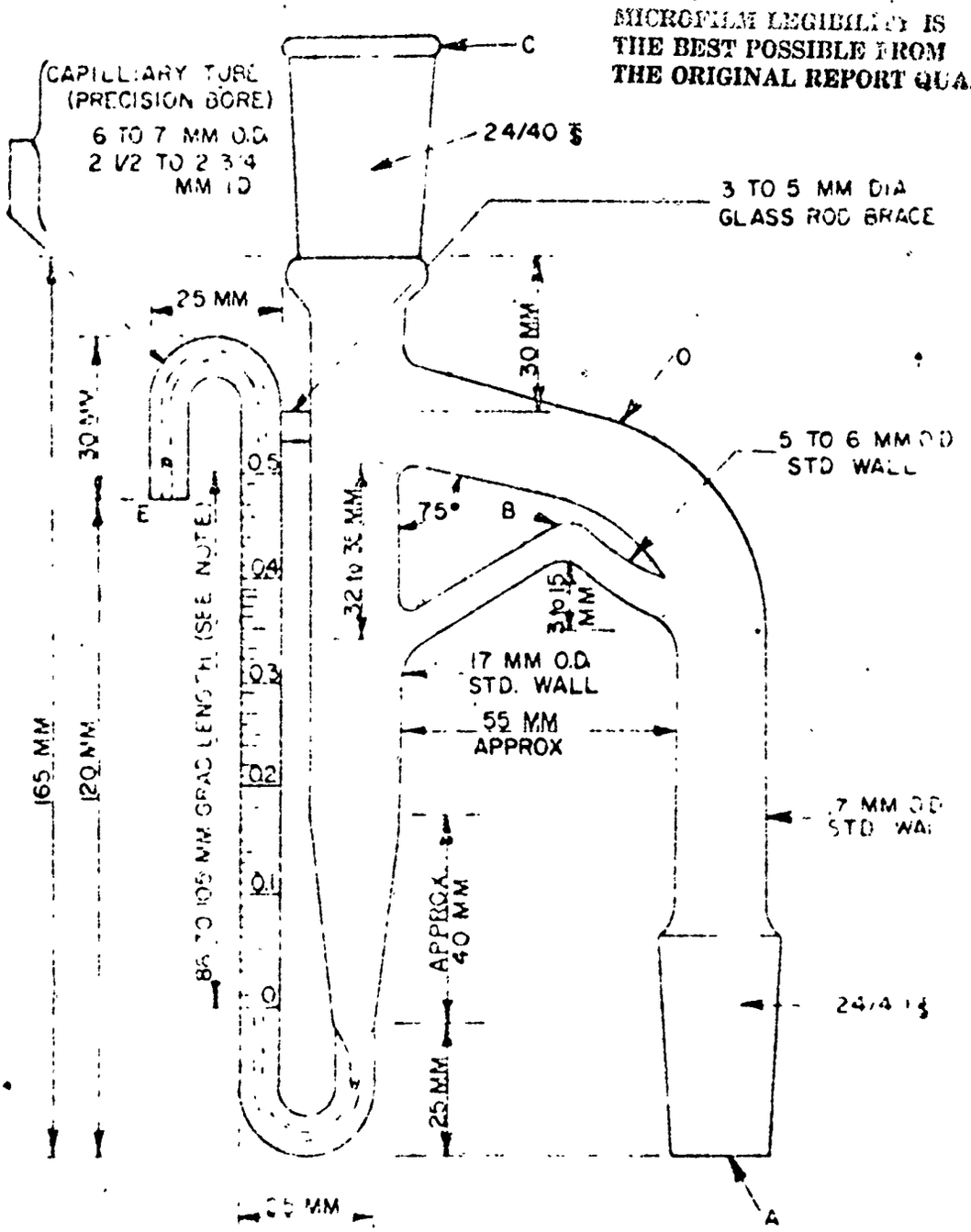

NOTE CAPACITY OF GRADUATED LENGTH OF CAPILLIARY TUBE = 0.5 ML: PRINCIPAL GRADUATIONS IN TENTHS (SUBDIVIDED INTO HUNDREDTHS)

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FIGURE 2. MOISTURE TRAP