

MIL-E-255A**25 MAY 1963****SUPERSEDING****JAN-E-355****21 SEPTEMBER 1945**

MILITARY SPECIFICATION

ETHYL CENTRALITE (CARBAMITE)

This specification has been approved by the Department of Defense and is mandatory for use by the Departments of the Army, the Navy and the Air Force.

1. SCOPE

1.1 Scope. This specification covers one grade of ethyl centralite for use in propellants and Explosives.

1.2 Classification. The ethyl centralite shall be of the following class as specified (see 6.1).

Class 1 — Lump

Class 2 — Powder

Class 3 — Flake

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

FEDERAL

PPP-D-728 — Drums, Fiber.

PPP-D-729 — Drums, Metal, 55 Gallon (For Shipment of Non-Corrosive Material).

RR-S-366 — Sieves; Standard; Testing.

UU-S-48 — Sacks Shipping, Paper.

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JAN-P-112 — Packaging and Packing for Overseas Shipment Drums, Plywood (For Drums Whose Weight of Contents Does Not Exceed 200 Pounds).

STANDARDS

MILITARY

MIL-STD-105 — Sampling Procedures and Tables for Inspection by Attributes.

FSC 6810

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MIL-STD-109 — Inspection Terms and Definitions.

MIL-STD-129 — Marking for Shipment and Storage.

PUBLICATIONS**— ORDNANCE CORPS**

ORD-M608-11 — Procedures and Tables for Continuous Sampling by Attributes.

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

2.2 The following documents form 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.

UNIFORM FREIGHT CLASSIFICATION RULES

(Application for copies of these freight classification rules should be addressed to the Consolidated Classification Committee, 202 Chicago Union Station, Chicago 6, Ill.)

AMERICAN TRUCKING ASSOCIATION PUBLICATION

National Motor Freight Classification Rules and Container Specifications

(Application for copies should be addressed to the American Trucking Association 1424 16th Street N. W., Washington, D. C.)

3. REQUIREMENTS

3.1 **Material.** The material shall consist essentially of diethyl diphenyl urea, CO (NC₆H₅-C₆H₅), meeting the requirements of this specification.

3.2 **Solidification point.** The solidification point of the ethyl centralite shall be 71.0

degrees centigrade (9°C.) to 72.5°C. inclusive when tested as specified in 4.3.1.

3.3 **Melted material.** The ethyl centralite shall melt to a bright clear liquid, free from scum and deposit when tested as specified in 4.3.2.

3.4 **Volatile content.** The volatile content of the ethyl centralite shall be 0.1 percent maximum (max.) when tested as specified in 4.3.3.

3.5 **Ash content.** The ash content of the ethyl centralite shall be 0.1 percent max. when tested as specified in 4.3.4.

3.6 **Secondary and tertiary amines.** The sum of the secondary and tertiary amines calculated as ethylaniline and diethylaniline, respectively, shall not exceed 0.20 percent when tested as specified in 4.3.5.

3.7 **Acidity.** The acidity of the ethyl centralite shall not exceed .04 percent when calculated as hydrochloric acid as specified in 4.3.6.

3.8 **Hydrolyzable chlorine compounds content.** The maximum hydrolyzable chlorine compounds content of the ethyl centralite shall be 0.001 percent when determined as specified in 4.3.7.

3.9 **Particle form.** The ethyl centralite shall meet the following particle form requirements for the type specified in 4.3.8.

3.9.1 **Class 1, lump.** The ethyl centralite shall be supplied in lump form.

3.9.2 **Class 2, powder.** A minimum (min.) of 99.9 percent of the ethyl centralite shall pass through a U. S. Standard Number 80 sieve.

3.9.3 **Class 3, flake.** A min. of 99.9 percent of the ethyl centralite shall pass through a U. S. Standard Number 8½ sieve. The thickness of the flakes shall not exceed 0.025 inches.

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3.10 Workmanship. The ethyl centralite shall be free from grit, visible impurities and foreign matter.

4. QUALITY ASSURANCE PROVISIONS

4.1 General quality assurance provisions. The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. 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. Reference shall be made to Standard MIL-STD-109 in order to define the terms used herein. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.

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 referred to herein, he shall submit a written description of the system to the contracting officer 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 provisions required by the provisions of 4.2 and 4.3 and 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 procedure. 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 changes to the written procedure that effects 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 products 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 from all inspection performed.
- (d) Specification number and date, together with an identification and date of changes.
- (e) Certificates of analysis on all material covered by referenced Government specifications procured directly by the contractor.
- (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 Government verification. Using the contractor's written quality assurance procedure (see 4.1.1), this detailed specification, and other contractual documents as a guide, the Government inspector shall verify all quality assurance operations performed by the contractor. Verification shall be in accordance with (a) or (b) as applicable, the decision being the responsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all quality assurance provisions of other specifications referenced in any of the contractual documents have been complied with. Deviations from prescribed or agreed upon procedures discovered by the Government inspector shall be brought to the attention of the supplier. Disposition of the product and remedial action shall be directed by the Government inspector and, depending on the nature of the deviation, may consist of lot rejections, screening, re-sampling, re-instruction of the supplier's employees, or other appropriate action:

- (a) Verification at the point of manufacture shall be accomplished at unscheduled intervals in accordance with 4.1.3.1 and 4.1.3.2.
- (b) Verification at the point of delivery shall be in accordance with 4.1.3.2.

4.1.3.1 Surveillance. Surveillance shall include, but is not limited to:

- (a) Observation of procedures concerning lot formation and identification.
- (b) Observation of sampling procedures and application of acceptance criteria.
- (c) Determination that all required examinations and tests are performed in accordance with the prescribed procedures of this specification, or approved equivalents thereto.

- (d) Review of procedures for control and disposition of non-conforming material.

4.1.3.2 Product inspection. Product inspection shall consist of Government inspection of product which has been previously inspected by the contractor and found to meet the quality assurance provisions of this specification. The inspection by the Government shall be performed in order to determine that the product is of the quality required by this specification and that the contractor's records are reliable.

4.2 Inspection provisions.

4.2.1 Lot information. A lot shall consist of one or more batches of ethyl centralite, produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each batch shall consist of that quantity of ethyl centralite that has been subjected to the same unit chemical or physical process intended to make the final product homogeneous.

4.2.2 Examination. Sampling plans and procedures for the following classification of defects shall be in accordance with standard MIL-STD-105. Continuous sampling plans, in accordance with Handbook ORD-M608-11 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 the individual characteristics listed using an AQL of 0.25 percent for each major defect and an AQL of 0.40 percent for each minor defect.

4.2.2.1 Drum, prior to filling (Polyethylene liner).

Categories	Defects	Method of inspection
Critical:	None defined.	
Major:	AQL 0.25 percent	
	101. Liner cut, torn or punctured	Visual
Minor:	None defined.	

MIL-E-255A**4.2.2.2 Drum, prior to filling (Polyethylene method).**

Categories	Defects	Method of Inspection
Critical:	None defined.	
Major:	AQL 0.25 percent	
	101. Laminations or coatings incomplete	Visual
Minor:	None defined.	

4.2.2.3 Drum, prior to closing (Polyethylene liner).

Categories	Defects	Method of Inspection
Critical:	None defined.	
Major:	AQL 0.25 percent	
	101. Liner not completely sealed	Visual
Minor:	None defined.	

4.2.2.4 Drum, sealed (Steel).

Categories	Defects	Method of Inspection
Critical:	None defined.	
Major:	AQL 0.25 percent	
	101. Weight of Contents	Scale
	102. Closing ring, damaged, or not properly positioned so it doesn't close drum properly	Visual
Minor:	AQL 0.40 percent	
	201. Marking misleading or unidentifiable	Visual

4.2.2.5 Drum, sealed (fiber), and sacks, paper.

Categories	Defects	Method of Inspection
Critical:	None defined.	
Major:	AQL 0.25 percent	
	101. Weight of contents	Scale
	102. Closure incomplete or damaged to extent that contents slip out	Visual
Minor:	AQL 0.40 percent	
	201. Markings misleading or unidentifiable	Visual

4.2.2.6 Drum, sealed (plywood).

Categories	Defects	Method of Inspection
Critical:	None defined.	
Major:	AQL 0.25 percent	
	101. Strapping missing or broken	Visual
	102. Weight of contents	Scale

Categories	Defects	Method of Inspection
Minor:	AQL 1.00 percent	
	201. Strapping mislocated	Visual
	202. Markings misleading or unidentifiable	Visual

4.2.3 Testing.

4.2.3.1 Determination of chemical and physical properties (see 3.2-3.9). Samples for these determinations shall be selected as specified herein.

4.2.3.1.1 Sampling by lot. A random sample of 10 containers shall be selected from each lot. When lots are comprised of 10 containers or less, each container shall be sampled.

4.2.3.1.2 Preparation of composite. Approximately two ounce primary sample of ethyl centralite shall be removed from each of the ten containers in order to equal twenty ounces and if there are less than 10 containers, equal primary samples in sufficient quantity to equal twenty ounces shall be removed from each container. The individual primary samples shall then be combined in order to form a homogeneous composite sample of twenty ounces and subjected to the tests specified in 4.3. If the composite sample fails to comply with any of the requirements specified, the lot shall be rejected.

4.3 Test methods and procedures.

4.3.1 Solidification point. Melt approximately 50 grams (gm.) of the sample in a covered 250 milliliters (ml.) beaker by immersing the beaker in a water bath at 90° to 95°C. for 15 minutes. The inner tube of the solidification apparatus shown on figure 1 shall be filled to within one and one half inches of the top with the molten ethyl centralite. The stirrer and thermometer shall be placed in the tube and the tube and the contents placed in a water bath at 85° to 90°C. for five minutes. The tube shall be removed from the water bath and placed in the apparatus. The molten material shall be stirred constantly, making approximately one up and

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down motion of the stirrer per second. Using a 76 millimeter partial immersion thermometer (American Standard Testing Method number 93C.) with a range of 60 – 90°C., the temperature of the molten material shall be recorded every 30 seconds until solidification begins as indicated by the temperature of the sample rising after it has reached a minimum. The stirring shall be continued until the temperature reaches a maximum.

4.3.2 Melted material. The appearance of the melted material shall be determined visually for compliance with the requirement specified herein. The examination shall be made during the test specified in 4.3.1.

4.3.3 Volatile matter. A weighed portion of approximately five (gm.) of the sample shall be transferred to a tared shallow dish, heated for two hours at 60°C., cooled in a desiccator and weighed. The loss in weight shall be calculated as percentage of volatile matter in the sample.

4.3.4 Ash. A weighed portion of approximately five gm. of the sample shall be transferred to a tared porcelain or silica crucible and sufficient alcohol then added to cover the material. The mixture shall be ignited, allowed to burn down slowly and then heated to redness until all carbon has been removed. Then, the mixture shall be cooled in a desiccator and weighed. The gain in weight shall be calculated as percentage of ash in the sample.

4.3.5 Secondary and tertiary amines.

4.3.5.1 Secondary amines. A weighed portion of approximately 25 gm. of the sample shall be transferred to an Erlenmeyer flask and dissolved with 30 ml. of benzene. Five ml. of an approximately 0.25 Normal (N) solution of acetic anhydride in xylene shall be added. The flask shall then be stoppered and allowed to stand overnight. Twenty-five ml. of 0.1N sodium hydroxide solution shall

be added, shaken thoroughly and transferred to a separatory funnel. The flask shall be washed with distilled water and the washings added to the contents of the funnel. Twenty-five ml. of 0.1N hydrochloric acid shall be added, shaken and the aqueous layer then separated. The funnel shall be washed with successive quantities of distilled water, adding the washings to the separated aqueous layer. The aqueous layer shall be titrated with 0.1N sodium hydroxide solution using the phenolphthalein as an indicator. At the same time, a blank determination shall be run through the complete procedure. The percentage of ethylaniline shall be calculated as follows:

$$\text{Percent ethylaniline} = \frac{12.1 \text{ N B} - (\text{V} - \text{C})}{\text{W}}$$

where:

B = ml. of sodium hydroxide solution required for blank.

V = ml. of sodium hydroxide solution required for sample (see 4.36).

C = ml. sodium hydroxide required for acidity of the sample.

N = normality of sodium hydroxide solution.

W = weight of sample.

4.3.5.2 Tertiary amines. Five gm. of the sample shall be dissolved in 20 ml. of benzene contained in a separatory funnel. Twenty ml. of 0.5 hydrochloric acid shall be added and the mixture shaken thoroughly. Two ml. of 1.0 sodium nitrate solution and sufficient water shall be added to the separated aqueous extract to make the total volume up to 50 ml. The solution shall be thoroughly mixed and allowed to stand for 30 minutes. By means of a colorimeter, the color of the test liquid shall be compared with that given in a blank determination in which 0.0010 gm. of diethylaniline (0.02 percent on a five gm. sample) is present. Calculate the percentage of diethylaniline in the sample (see 6.2).

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4.3.6 Acidity. A weighed portion of approximately 25 gm. of the sample shall be placed in a flask and melted with 30 ml. of hot distilled water. The mixture shall be shaken vigorously until the ethyl centralite crystalizes. The mixture shall be cooled and titrated with 0.1N sodium hydroxide solution, using phenolphthalein as an indicator. At the same time, a blank determination shall be run and the volume of sodium hydroxide used in titrating the sample corrected. Acidity shall be calculated as hydrochloric acid as follows:

$$\text{Hydrochloric Acid Percent} = \frac{3.65 \text{ VN}}{W}$$

where:

V = volume of Sodium Hydroxide used in titration.

N = normality of the alkali.

W = weight of sample in gms.

4.3.7 Hydrolyzable chlorine compounds. A five gm. portion of the sample shall be transferred to a 150 ml. beaker. Ten ml. of a five percent solution of sodium ethylate in alcohol shall be added and the solution heated on a steam bath for 15 minutes. Seventy-five ml. of distilled water shall be added to the contents of the beaker and sufficient time allowed for the ethyl centralite to crystalize. The solution shall be made acid to litmus with concentrated nitric acid and then filtered through a Number 41 Whatman filter paper, or equivalent, into a 100 ml. Nessler tube. If not clear, the solution shall be refiltered. The beaker and filter paper shall be washed with distilled water until the Nessler tube attains a volume of 100 ml. Five ml. to 10 percent silver nitrate shall be added, mixed thoroughly, and the solution shall be allowed to stand for one-half hour. Concurrent with the analysis, standard solutions (containing 0.001 percent and 0.002 percent sodium chloride) shall be prepared as follows: 1.650 ± 0.005 grams of reagent grade sodium chloride shall be transferred to a one liter volumetric flask. The flask shall be filled to the mark

with distilled water and mixed well. A 10 ml. aliquot shall be transferred to a second one liter volumetric flask, and also, a 20 ml. aliquot shall be transferred to another one liter volumetric flask, brought to the mark with distilled water and mixed well. One hundred ml. of each chloride standard shall be transferred to separate Nessler tubes. A third tube shall contain 10 ml. of distilled water as a blank. Each standard solution in the Nessler tubes shall be acidified (to litmus paper) with concentrated nitric acid, and 5 ml. of 10 percent silver nitrate solution shall be added to each tube, mixed well, and allowed to stand for 30 minutes. The turbidity of the sample mixture shall be compared with the standards by viewing downward against a well lighted white background.

4.3.8 Particle form.

4.3.8.1 Screen test (Class 2 only). A bottom pan shall be fitted to a U. S. Standard Number 30 sieve conforming to Specification RR-S-366. A weighed portion of 100 ± 2 gm. of the sample shall be placed on the sieve, covered, and shaken for 10 minutes by hand or five minutes by means of a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. The amount retained on the sieve (if any) shall be weighed and calculated in terms of percentage of material passing through the sieve.

4.3.8.2 Screen test (Class 3 only). The procedure in 4.3.8.1 shall be followed except the screen shall be a U.S. Standard Number 3½.

4.3.8.2.1 Particle thickness. The average maximum thickness of 25 flakes shall be determined by means of a micrometer.

5. PREPARATION FOR DELIVERY

5.1 "Preservation and packaging — Not applicable".

MIL-E-255A**5.2 Packing.**

5.2.1 *Level A.* Unless otherwise specified in the contract or purchase order, Ethyl Centralite (Carbamite) shall be packed in a fiber, plywood or metal drum in accordance with Specifications PPP-D-728 (Type III, Grade A), JAN-P-112 and PPP-D-729 (Type III, Full Removable Head Type), respectively. Fiber and plywood drums shall be furnished with a nominal 0.004 inch thick polyethylene liner properly heat sealed or otherwise closed to afford protection to the contents equivalent to that given by a heat seal closure. (Alternatively, an equivalent degree of protection may be furnished by suitable laminations or coatings of the inner surface of the drum). Alternatively, Ethyl Centralite (Carbamite) may be packed in paper shipping sack in accordance with Construction No. 14X Specification UU-S-48. Closure of drums or sacks shall be in accordance with the applicable specification.

5.2.2 *Level C.* Ethyl Centralite (Carbamite) shall be packed to afford protection against damage during direct shipment from the supply source to the first receiving activity for immediate use. Containers shall comply with Uniform Freight Classification Rules and Container Specifications for rail shipments or National Motor Freight Rules and Container Specifications for truck shipments, as applicable.

5.3 **Marking.** In addition to any special marking required by the contract or purchase

order, containers shall be marked in accordance with Standard MIL-STD-129.

6. NOTES

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

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

(b) Type of ethyl centralite required.

6.2 A Du Boc or similar type colorimeter may be used (see 4.3.5.2).

6.3 **Intended Use.** Ethyl centralite is intended for use as a stabilizer in propellants.

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 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.

Custodians:

Army—Ordnance Corps.

Navy—Bureau of Naval Weapons

Preparing activity:

Army—Ordnance Corps.

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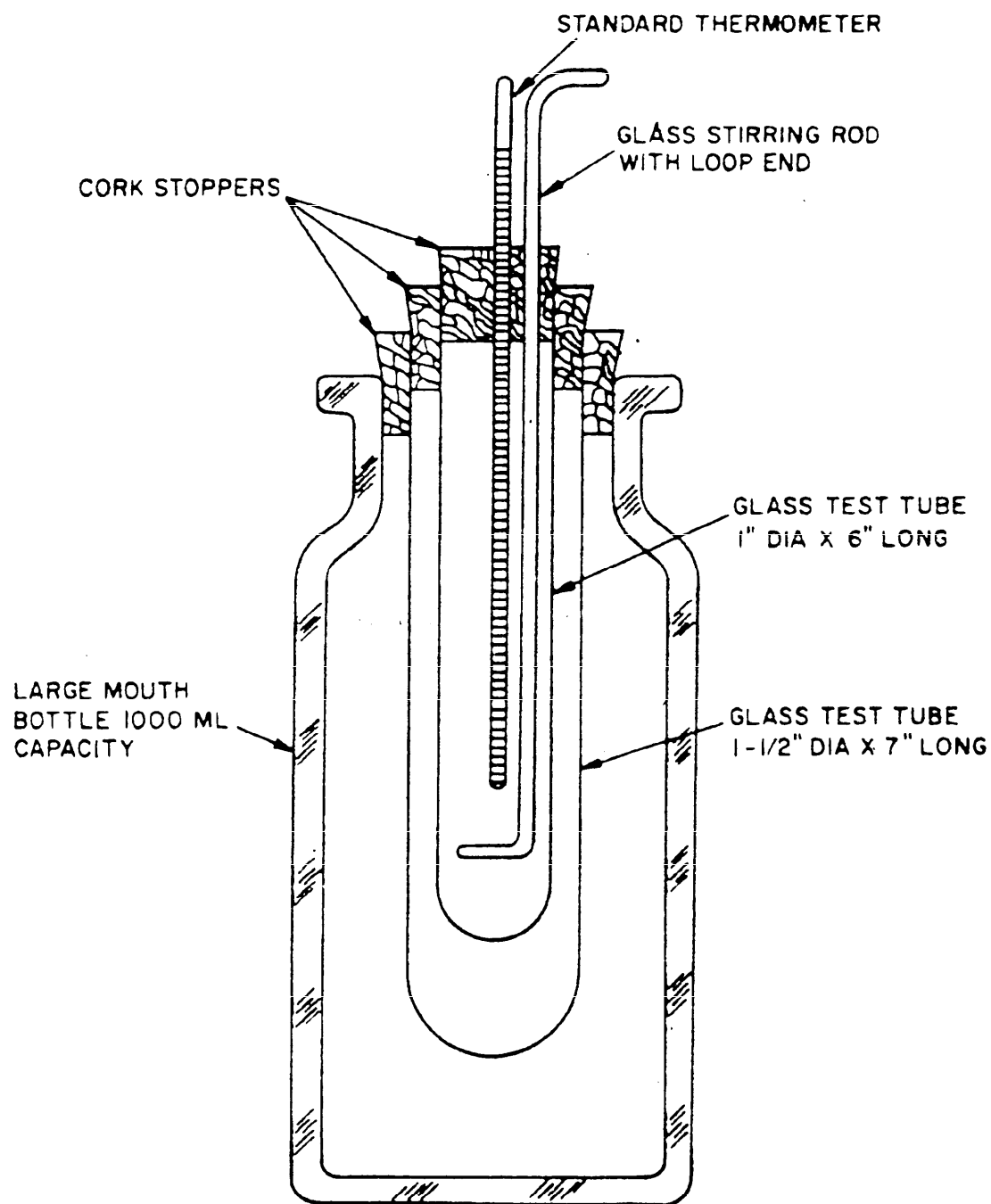


FIGURE I. Solidification point apparatus