

MIL-E-9500B
 15 June 1983
 SUPERSEDING
 MIL-E-9500A
 21 JUNE 1968

MILITARY SPECIFICATION

ETHYLENE GLYCOL, TECHNICAL

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 This specification covers technical grade ethylene glycol.

2. APPLICABLE DOCUMENTS

2.1 Government documents. Unless otherwise specified, the following specifications, standards, and handbooks of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

Specifications

Federal

PPP-B-636	Box Fiberboard
PPP-C-569	Containers, Plastic, Molded (For Liquids, Parts, and Powders)
TT-W-572	Wood-Preservative; Water-Repellent

Military

MIL-P-15011	Pallets, Material Handling, Wood, Post Construction, 4-Way Entry
MIL-D-43703	Drum, Molded Polyethylene

Standards

Federal

FED-STD-313	Material Safety Data Sheets, Preparation and the Submission of
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Military

MIL-STD-129	Marking for Shipment and Storage
MIL-STD-147	Palletized Unit Loads
MIL-STD-1188	Commercial Packaging of Supplies and Equipment

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: SA-ALC/SFTT/Kelly AFB TX 78241 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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

2.2. Other Publications. The following document(s) form a part of this specification to the extent specified herein. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM STANDARDS

D92	Test for Flash and Fire Points by Cleveland Open Cup.
D891	Test for specific gravity of industrial aromatic hydrocarbons and related materials
D1078	Test for distillation range of volatile organic liquids
D1193	Specification for reagent water
D1209	Test for color of clear liquids (platinum-cobalt scale)
E202	Analysis of ethylene glycols and propylene glycols
E203	Test for water using Karl Fischer Reagent.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA. 19103)

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

3. REQUIREMENTS

3.1 Chemical requirements. The chemical requirements of the ethylene glycol shall be as specified in Table I.

TABLE I. CHEMICAL REQUIREMENTS

<u>Requirements</u>	<u>By Weight</u>	<u>Test</u>
Chlorides, no precipitate Present, by Test	----	4.3.1
Total glycols, minimum	99.5%	4.3.2
Water, maximum	0.5%	4.3.3
Acidity (Calculated as acetic acid), maximum	0.01%	4.3.4
Ash residue max per 100 mililiters	0.005 grams	4.3.5

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Distillation	
Initial boiling point, 192°C minimum	4.3.6
95% distilled, 200°C maximum	
Dry point, 208°C maximum	
Water solubility, miscible with water in all proportions at 25°C (77°F)	4.3.7

3.2 Physical requirements. The physical requirements shall be as specified in Table II.

TABLE II. PHYSICAL REQUIREMENTS

<u>Requirements</u>	<u>Tests</u>
Color, not more than 15 on the platinum-cobalt scale	4.3.8
Flash point 116°C(240°F) minimum	4.3.9
Odor, mild, not be objectionable	4.3.10
Specific gravity, 1.1151 to 1.1156 at 20°/20°C	4.3.11
Appearance	4.3.12

4. QUALITY ASSURANCE PROVISIONS

4.1 Lot. A lot will consist of the material produced by one manufacturer in not more than a 24-hour period under essentially the same manufacturing conditions and with no change of materials, providing the operation is continuous and submitted for inspection at the same time. In the event the process is a batch operation, each batch will constitute a lot.

4.2 Sampling.

4.2.1 Sampling shall be conducted in accordance with Table III. Agitate the contents of each container to be sampled. A one quart representative sample shall be taken from each container, and these shall be combined together to form a composite sample for analysis. Each composite sample shall be placed in a separate, clean, dry container labeled to identify the lot and containers from which it was taken. The total composite sample from each lot shall be no less than 1 gallon.

TABLE III. SAMPLING FOR TEST

Number of Containers in lot or batch.	Number of sample containers
2 - 25	2
26 - 150	3
151 - 1,200	5
1,201 - 7,000	8
7,001 - 20,000	10
over 20,000	20

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4.2.2 Inspection Procedure. Each composite sample shall be tested separately as specified in 4.3.

4.3 Tests. Water in accordance with ASTM D1193 and reagent grade chemicals shall be used for testing. Blanks shall be run and corrections applied when significant. Tests shall be conducted as follows:

4.3.1 Chlorides. Place 10 milliliters (ml) of the material in a 30 ml test tube, Add 10 ml of water and mix. Add 10 drops of concentrated nitric acid, agitate the mixture, and filter. Add 10 drops of a 3 percent aqueous solution of silver nitrate to the filtrate. No precipitate shall settle out within 30 minutes after the addition of silver nitrate solution. A slight turbidity will not be caused for rejection.

4.3.2 Total Glycols. Total glycols shall be determined in accordance with ASTM E202.

4.3.3 Water Content. Water content shall be determined in accordance with ASTM E203 except 10 to 40 grams of sample, depending on the anticipated moisture content, shall be used.

4.3.4 Acidity. Acidity shall be determined by measuring 108 ml of sample in a graduate and transferring it to a 250 ml Erlenmeyer flask. Five drops of 1.0 percent alcoholic solution of phenolphthalein indicator shall be added and the sample titrated with standard 0.1N alcoholic KOH to the first pink end point permanent for 15 seconds. The calculation is as follows:

$$\text{ml KOH} \times 0.005 = \text{acidity, percent by weight as acetic acid}$$

4.3.5 Ash residue. Ash residue shall be determined by measuring 50 ml of the sample in a graduate and transferring to a 125 ml platinum dish which has been ignited to constant weight, cooled in a desiccator, and tared to the nearest 0.1 milligram. The dish shall be heated until the vapors continue to burn after the flame is withdrawn. The combustion shall be protected from drafts and the vapors allowed to burn spontaneously until the liquid is consumed. The dish shall then be ignited to a dull red heat, allowed to cool in a desiccator and weighted to the nearest 0.1 mg. The ash residue per 100 ml shall be calculated as follows:

$$\text{gm ash/100 ml} = (\text{gm residue}) \times 2$$

4.3.6 Distillation. Distillation shall be conducted according to ASTM D1078 and E202, except that distillation shall be conducted at a pressure of 760 millimeters (mm) of mercury or corrected thereto by adding 0.043°C for each mm under 760 mm or subtracting from every mm over 760 mm.

4.3.7 Miscibility with water. Miscibility with water shall be determined by transferring 25 ml of the sample to 25°C to a 100 ml glass stoppered graduate and adding 25 ml of water at 25°C in 5 ml portions, shaking the graduate well after each addition. Twenty-five ml of the sample shall be added to 25 ml of water in the same manner. If there is no cloudiness, stratification, or turbidity at any time, the sample shall be considered completely miscible.

4.3.8 Color. Color shall be determined in accordance with ASTM D1209 and E202.

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4.3.9 Flash point. The flash point of the material shall be determined according to ASTM D92, except that the fire point need not be determined.

4.3.10 Odor. Odor shall be determined by pouring 5 ml of the sample on clean filter paper and observing the odor immediately.

4.3.11 Specific gravity. Specific gravity of the material shall be determined for 20⁰/20⁰C according to ASTM D891 and E202.

4.3.12 Appearance. Appearance shall be clear, and material must be free of suspended matter when examined visually by transmitted light.

5. PREPARATION OF DELIVERY

5.1 Packaging. Packaging shall be Level A. The container used shall be compatible with ethylene glycol and shall not show any interior deterioration during storage. Plastic containers with a nominal capacity of 1 or 55 gallons, as specified by the procuring activity, shall be used for packaging ethylene glycol.

5.1.1 Fifty-five Gallon Drums. Fifty-five gallons of ethylene glycol shall be packaged in drums conforming to size 4 of MIL-D-43703.

5.1.2 One Gallon Containers. One gallon of Ethylene Glycol shall be packaged in the rectangular plastic container, with tamper proof seal and screw cap, normally used in commercial activity and composed of material that will not affect nor be affected by the contents.

5.2. Packing. One gallon containers shall be packed level A or industrial as specified (See 6-2). The 55 gallon containers shall require no further packing.

5.2.1 Level A. Six one gallon containers shall be packed in a snug fitting fiberboard box conforming to Type CF, Class-Weather Resistant, Variety SW, Grade V3c of PPP-B-636. Each container shall be fitted with full height partitions of the same material as used in construction of the box. If necessary to protect plastic bottles from contact with staples used in box construction full height liners and top and bottom fiberboard pads shall be used.

5.2.2 Industrial. Ethylene Glycol packaged as specified in 5.1 shall be packed in accordance with the provisions of MIL-STD-1188 except for marking which shall comply with 5.5.

5.3 Palletization. When specified in the contract or order Ethylene Glycol packed in accordance with MIL-STD-147.

5.4 Preservative treatment. Grade A pallets, or the finished wood parts thereof, shall be completely immersed for a period of three minutes in a water-repellent wood preservative complying with type I or II, composition B, of TT-W-572.

5.5 Markings. In addition to any special markings required by the contract or order, unit packages, intermediate packages, and shipping containers shall be marked in accordance with MIL-STD-129.

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5.5.1 All containers shall be marked with the following additional information:

ETHYLENE GLYCOL
WARNING! POISONOUS IF SWALLOWED.
DO NOT TAKE INTERNALLY!

5.6 Material Safety Data Sheet. A material safety data sheet shall be furnished as required by FED-STD-313.

6. NOTES

6.1 Intended Use. The ethylene glycol covered by this specification is intended for use as a heat-transfer of fluid.

6.2 Ordering data. Procurements should specify the followings:

- (a) Title, number, and date of this specification.
- (b) Type and size of container required (see 5.1).
- (c) Level of packaging and packing required.
- (d) Special markings required.
- (e) Palletization required.

6.3 The ethylene glycol should be purchased by volume, the unit being one US gallon of 231 cubic inches at 15.0°C (59.0°F).

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