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SUPERSEDING
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MILITARY SPECIFICATION

PROPANEDIOL, 1, 2 (METRIC)

This specification is approved for use by the Naval Sea Systems Command, Department of the Navy and is available for use by all Departments and Agencies of the Department of the Defense.

1. SCOPE

1.1 Scope. This specification covers one type of propanediol, 1, 2 hereinafter referred to as "propylene glycol".

2. APPLICABLE DOCUMENTS

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

STANDARDS

MILITARY

MIL-STD-105	Sampling Procedures and Table for Inspection by Attributes
MIL-STD-129	Marking for Shipment and Storage
MIL-STD-1218	ACS Chemicals

(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).

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Ordnance Station, Standardization/Documentation Division (501), Indian Head, MD 20640, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

2.2 Other publications. 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 or request for proposal shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D-86- 78	Distillation of Petroleum Products, Test for
ASTM D-270- 65	Sampling Petroleum and Petroleum Products
ASTM D-1209- 79	Color of Clear Liquids (Platinum Cobalt Scale)
ASTM E-1- 76	ASTM Thermometers, Specification for

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

UNIFORM CLASSIFICATION COMMITTEE AGENT

Uniform Freight Classification Rules

(Application for copies should be addressed to the Uniform Classification Committee, Room 1106, 222 South Riverside Plaza, Chicago, IL 60606).

3. REQUIREMENTS

3.1 Physical and chemical requirements. The physical and chemical properties of the propylene glycol shall conform to the limits specified in TABLE I when tested as specified herein. If the lot fails to conform to any of the requirements specified, the lot shall be rejected.

TABLE I. Physical and chemical requirements.

Property	Requirements Minimum	Requirements Maximum	Test Method
Color (Pt-Co Scale)	-	10	4.3.2.1
Specific gravity at 25°/25°C	1.0340	1.0390	4.3.2.2
Acidity (as acetic acid) (% by wt)	-	0.03	4.3.2.3
Refractive index (n_D25°C)	1.4300	1.4340	4.3.2.4
Distillation range, 185° to 190°C at atmospheric pressure (%)	98	-	4.3.2.5
Carbonyl (ppm)	-	40	4.3.2.6
Moisture (% by wt)	-	0.50	4.3.2.7
Dipropylene glycol (%)	-	0.10	4.3.2.8

3.2 Workmanship. The propylene glycol shall be a water white, homogeneous liquid, free from dirt, sediment, and other suspended foreign matter when examined visually by transmitted light.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Inspection provisions.

4.2.1 Lot formation. A lot shall consist of the propanediol, 1, 2 offered for acceptance at one time which has been produced by one manufacturer, at one plant, from the same materials, and under essentially the same manufacturing conditions provided the operation is continuous. In the event the process is a batch operation, each batch shall constitute a lot (see 6.3).

4.2.2 Sampling. Sampling from tank cars shall be conducted in accordance with ASTM D-270. Sampling from smaller containers shall be conducted as follows. A random sample shall be taken from each lot in accordance with MIL-STD-105, Inspection Level S-2, Acceptance number zero. If there are fewer than three containers in a lot, each container shall be sampled. In all other cases, no fewer than three containers shall be selected. A 500 milliliter (mL) specimen shall be removed from each container in the sample and placed in a clean, dry container. The container shall be labeled to identify the lot and container from which it was taken.

4.2.3 Test specimens. A composite specimen shall be made with equal portions from each specimen, and the composite specimen shall be tested as specified in 4.3. If there are fewer than three specimens, each one shall be tested as specified in 4.3.

4.3 Quality conformance inspection. The material shall be subjected to all the following inspections and tests for acceptance. When specified in the contract (see 6.2), the supplier shall submit a report giving the results obtained for all inspections and tests performed and a certified statement that the lot meets all the requirements of this specification. Unless otherwise specified, all chemicals shall be ACS grade in accordance with MIL-STD-1218 and distilled water shall be used. Where applicable, blank determinations shall be run and corrections applied where significant.

4.3.1 Visual inspection. All samples shall be visually inspected to determine conformance to the workmanship requirements of 3.2.

4.3.2 Test methods and procedures.

4.3.2.1 Determination of color. The color of the propanediol 1, 2 shall be determined in accordance with ASTM D-1209, platinum cobalt scale.

4.3.2.2 Determination of specific gravity. Determine the specific gravity at 25°/25° C by any convenient method that is accurate to the fourth decimal place.

4.3.2.3 Determination of acidity. Measure 100 mL of ethyl alcohol into a flask, add three drops of phenolphthalein indicator, and titrate the alcohol to a faint pink color with 0.1N sodium hydroxide solution. Add an accurately weighed 30 to 40-gram (g) portion of the sample to the alcoholic solution. Titrate the mixture with 0.1N sodium hydroxide. Calculate the percentage of acidity as acetic acid as follows:

$$\text{Percent acetic acid} = \frac{6.005\ VN}{G}$$

where: V = sodium hydroxide required for titration, mL
N = normality of sodium hydroxide solution
G = weight of sample, g

4.3.2.4 Determination of refractive index. The following procedure shall be used for the determination of refractive index using a Bausch and Lomb Abbe refractometer or equivalent:

a. Wipe with caution to preclude scratching the prism surface of the refractometer with a tissue wet with methanol or xylene, and then with a dry tissue. Adjust the temperature of the water bath and the velocity of the circulating fluid so that the thermometer reads 25.0° ± 0.1°C.

b. Rotate the body of the instrument and the moving arm away from the operator until the interface between the prisms is horizontal. Loosen the prism clamp and drop the lower prism. With a glass stirring rod, add a few drops of the sample liquid to be tested to the lower prism, bring the prism faces together, and lock the prism clamp. Rotate the instrument to the normal viewing position. Adjust the mirror to reflect light into the telescope.

c. Unclamp the index arm and move it to the far end of the scale. Focus the telescope eyepiece sharply on the crosshairs. Bring the divided field into view by moving the index arm forward. The field may have a colored border, which can be achromatized by use of the compensators rotated by a pinion. The field will be sharpest when the edge is just turning blue. Bring the field almost into coincidence with the crosshairs by moving the index arm. Adjust the mirror for the maximum illumination of the field. Obtain the final adjustment of the field line to the crosshairs by means of the fine adjustment screw.

d. Focus the magnifier on the scale and rotate it so that the scale is well illuminated. Read the scale directly to three decimal places and estimate the figure in the fourth decimal place.

e. If the index reading does not remain constant within one unit in the fourth decimal place for 1 minute, it may indicate diffusion of solvent remaining on the prisms or insufficient time for thermal equilibrium. Place a new sample of the liquid to be tested on the prism, taking care that the prism is clean and dry. Sufficient time should elapse after placement of the drop to insure that the liquid film is at the prism temperature.

f. Report the reading obtained as $n_D 25°$ C.

4.3.2.5 Determination of distillation range.

4.3.2.5.1 Apparatus. Use the distillation apparatus described in ASTM D-86 with the following exceptions:

a. Thermometer. Use an ASTM Partial Immersion Thermometer having a range of -5° to + 300° C and conforming to the requirements for thermometer 2C as prescribed in ASTM E-1 or a thermometer having a range of 95° to 225° C and conforming to the requirements for thermometer 42C as prescribed in ASTM E-1.

b. Condenser. A Liebig glass condenser 560 millimeters (mm) in length with 400 mm in contact with the cooling water may be used in place of the bath-type condenser.

4.3.2.5.2 Procedure. With the receiving graduate, transfer exactly 100 mL of the sample directly into the flask, allowing none to run into the side tube and allowing the graduate to drain thoroughly. If the sample contains dissolved or suspended water it is advisable to add a few small pieces of pumice or broken glass to promote smooth distillation. Insert the thermometer so that the top of the mercury bulb (or the top of contraction chamber if the Solvents Distillation Thermometer is used) is level with the bottom of the side tube. Connect the side tube to the condenser, with the bottom of the flask resting securely in the opening in the asbestos board. Apply heat cautiously and regulate it so that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 minutes. Record as the initial boiling point with thermometer reading when the first drop falls from the end of the condenser. When the distillation begins, regulate the heat so that the distillate is collected at a rate of not less than 4 nor more than 5 mL/min (approximately 2 drops per second.) Observe and record the temperature when 5 mL have been collected in the receiving cylinder, and thereafter when the level of the distillate reaches each 10 mL division of the graduate, including a 95 mL reading. Discontinue the distillation when the temperature reaches that specified in the minimum percentage requirement, or whenever the temperature rise stops and the thermometer reading starts to fall, and record the maximum temperature reached. Allow the condenser to drain and record the percentage distilled.

4.3.2.6 Determination of carbonyl.

4.3.2.6.1 Equipment.

a. Beckman Expandomatic pH Meter or other pH meter capable of titration in the millivolt (mV) mode.

b. Glass electrode (Beckman 41263 or equivalent.)

c. Calomel reference electrode (Beckman 29400 or equivalent.)

d. 10-mL burette.

e. 15-mL pipette.

4.3.2.6.2 Reagents.

a. Hydroxylamine hydrochloride. Recrystallize the commercial product from a 2:1 alcohol-water solution. Wash the crystals with a small amount of alcohol and air dry.

b. Alcoholic potassium hydroxide. Add 3g of reagent potassium hydroxide to 1 liter of isopropyl alcohol. Bring to the boiling point and stir until solution is effected. Store in a container vented to the atmosphere through a guard tube containing soda lime or soda asbestos (ascarite) to protect from atmospheric carbon dioxide. Do not permit the solution to contact cork, rubber or saponifiable stopcock grease. Dispense in such a manner that only the clear top portion of the liquid is obtained.

4.3.2.6.3 Procedure. Weigh a 100g sample of the propylene glycol into a 250 mL beaker. Add 3.0 g of the recrystallized hydroxylamine hydrochloride, 10 mL of ethyl alcohol and 4.5 mL of distilled water. Stir with a magnetic stirrer until the hydroxylamine hydrochloride is dissolved and let stand 1 hour (h) at room temperature. Fill the 10-mL burette with the alcoholic potassium hydroxide solution. Using a Beckman expandomatic pH meter or an equivalent, titrate the sample potentiometrically. Equip the instrument with a glass electrode and calomel reference electrode. Prepare the electrodes in accordance with the manufacturer's instructions. Blot the electrodes with tissue and lower into the reaction mixture. Depress both the expanded and + mV push buttons. Stir the solution until equilibrium is attained. Use the "standardize" knob to move the needle to 180 mV. (In this mode the full scale of the meter registers 200 mV. For meters other than Beckman, operate in accordance with the manufacturer's instructions for mV titrations.) Add the potassium hydroxide solution in increments of 0.5 mL with the burette tip immersed in the solution and with constant stirring. Allow time for the reading to stablize and record the mV reading after each addition. The mV change for the entire titration should be about 100-120. The electrodes should be returned to water after each titration. Plot mL KOH solution vs mV reading. Draw the best curve through the points and take the inflection point as the end point. Standardize the KOH solution by titrating approximately 80mg potassium biphthalate to a phenolphthalein end point.

Calculations.

Normality of KOH solution

$$N = \frac{\text{mg potassium biphthalate}}{204 \times \text{mL KOH}}$$

Carbonyl

$$C = \frac{\text{mL KOH} \times 30 \times 1000 \times N}{\text{sample weight (g)}}$$

C = ppm carbonyl in the sample calculated as formaldehyde

4.3.2.7 Determination of moisture. To the titration flask add 75 to 100 mL of methanol. Titrate the methanol to a potentiometric end-point with stabilized Karl Fischer reagent. Transfer an accurately weighed sample of propylene glycol (5 to 15 g) to the titration flask. Titrate the sample to a potentiometric end-point. Calculate the moisture content as follows:

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

where: K = Karl Fischer reagent used in titration, mL
F = reagent factor (g of water per mL of reagent)
W = weight of propylene glycol, g

4.3.2.8 Determination of dipropylene glycol.

a. Equipment.

1. Gas Chromatograph with flame ionization detector
2. Recorder, 0-1 mV strip chart recorder
3. Auxiliary equipment for use in gas chromatography

b. Reagents.

1. Fluoropak 80, 40-70 mesh
2. Carbowax 20M
3. Chloroform, reagent grade
4. Dipropylene glycol, U.S.P.

c. Column preparation.

1. Prepare a column packing material of 5% Carbowax 20M on Fluoropak 80 by dissolving the carbowax in chloroform and coating by a suitable method. Use only the material which will pass a 40 mesh screen. Prepare a 6.4 mm x 152 cm stainless steel column using a mechanical vibrator to completely fill the tube. Condition the column for 2 h at 210°C.

d. Gas chromatographic conditions.

1. Oven temperature 210°C
2. Injector and manifold temperatures 210°C
3. Carrier gas-helium at 40 mL/min
4. Detector - flame ionization
5. Sample size - 2 microliters (µL)
6. Attenuation - to suit the substance being measured

e. Preparation of standards.

1. Prepare high purity propylene glycol for use as standards by placing about 600 mL of propylene glycol in a 1 L round bottom flask and fit with a short fractionating column. Distill the material at a reasonable rate. Discard the first 25 mL fraction and collect 200 mL.

2. Using the distilled propylene glycol fraction prepare two standards of 0.1% and 0.2% by weight added dipropylene glycol.

3. Obtain chromatograms of the distilled propylene glycol fraction and of each of the two standards. Since the dipropylene glycol can consist of three isomers, its chromatogram should show two major peaks and a smaller third one. Select an attenuation for the dipropylene glycol elution such that at least one of the peaks for the 0.2% standard is 25 mm or more in height. Determine the relative peak areas of the dipropylene glycol elutions by a "cut and weigh" method including all significant peaks after that of the propylene glycol. If the distilled propylene glycol shows an appreciable amount of dipropylene glycol (more than 0.01%) then this should be measured by a method of additions procedure and a corresponding correction made to the 0.1% and the 0.2% standards.

f. Procedure.

1. Plot the peak areas against percent dipropylene glycol. Obtain chromatograms of the samples to be analyzed using the conditions in d. and the procedures in e. above. Measure the peak area for the dipropylene glycol and determine its percent from the standard curve.

4.3.3 Rejection criteria. Failure of any sample to meet any requirement of this specificaton shall be cause to reject the lot or batch.

5. PACKAGING

5.1 Packaging.

5.1.1 Level C. The propylene glycol shall be packaged in uniform quantities for shipment in accordance with the manufacturer's commercial practice. Packages shall be of uniform size, shape, and material.

5.2 Packing.

5.2.1 Level C. The propylene glycol shall be packed in uniform quantities in such a manner as to assure carrier acceptance and afford protection against damage during direct shipment from the supply source to the first receiving activity for immediate use. Containers used shall comply with Uniform Freight Classification Rules or other carrier regulations applicable to the mode of transportation. Containers shall be of uniform size, shape, and material.

5.3 Marking. In addition to any special marking required by the contract, unit packages and shipping containers shall be marked in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. The propylene glycol covered by this specification is intended for use in the manufacture of propellant ingredients.

6.2 Ordering data. Procurement documents should specify the following:

6.2.1 Procurement requirements.

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

b. Whether tank cars or containers are to be used for shipment.

c. Size of containers required.

d. Whether a certified analysis and/or test reports are required.

6.2.2 Contract data requirements. The items of deliverable data which may be required by this specification are cited in 4.3.

Data Requirement	Applicable DID*
Test Report	DI-T - 2072
Certified Analysis	------

*DIDs (Data Item Descriptions/DD Form 1664) for the above data requirements are documented in the applicable ADL (Authorized Data List.) Such data will be delivered as identified on completed (numbered) DIDs when specified on DD Forms 1423 (Contract Data Requirements Lists) and incorporated into applicable contracts.

6.3 Batch. A batch is defined as that quantity of propylene glycol that has been subjected to the same unit chemical or physical process intended to make the final product homogeneous.

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DOCUMENT IDENTIFIER AND TITLE
DOD-P-82670(OS) PROPANEDIOL, 1, 2 (METRIC)

NAME OF ORGANIZATION AND ADDRESS	CONTRACT NUMBER
	MATERIAL PROCURED UNDER A ☐ DIRECT GOVERNMENT CONTRACT ☐ SUBCONTRACT

1. HAS ANY PART OF THE DOCUMENT CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?
   A. GIVE PARAGRAPH NUMBER AND WORDING.

   B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES

2. COMMENTS ON ANY DOCUMENT REQUIREMENT CONSIDERED TOO RIGID

3. IS THE DOCUMENT RESTRICTIVE?
   ☐ YES ☐ NO *(If "Yes", in what way?)*

4. REMARKS

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