

DOD-P-82671(OS)
 19 January 1979
 SUPERSEDING
 WS 2103
 30 July 1965

MILITARY SPECIFICATION

PROPYLENE GLYCOL DINITRATE

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 Defense.

1. SCOPE

1.1 Scope. This specification covers the requirements for one type of propylene glycol dinitrate (PGDN) for use in the manufacture of propellants.

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.

SPECIFICATION

MILITARY

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Propanediol, 1, 2

STANDARDS

MILITARY

MIL-STD-129

Marking for Shipment and Storage

MIL-STD-286

Propellants, Solid: Sampling, Examination and Testing

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.

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2.2 Other publications. The following document forms 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.

CODE OF FEDERAL REGULATIONS

49 CFR 171- 190

Transportation

(The Interstate Commerce Commission Regulations are now a part of the Code of Federal Regulations (1975 Edition and Revisions) available from the Superintendent of Documents, Government Printing Office, Washington, DC 20401. Orders for the above publication should cite "49 CFR 100-199 (latest revision)").

(Technical society and technical 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 Material. The propylene glycol dinitrate shall be a product made from propanediol, 1, 2 which conforms to the requirements of DOD-P-82670.

3.2 Physical and chemical requirements. The physical and chemical properties of the propylene glycol dinitrate 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		Test method
	Minimum	Maximum	
Nitrogen (% by wt)	16.82	16.92	4.3.2
Acidity (as sulfuric acid) (% by wt)	-	0.001	4.3.3
Alkalinity (as sodium carbonate) (% by wt)	-	0.001	4.3.3
Moisture (% by wt)	-	0.50	4.3.4
Stability (potassium iodide at 82°C) (minutes)	10	-	4.3.5

3.3 Workmanship. The propylene glycol dinitrate shall be 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 supplies and services conform to specified requirements.

4.2 Inspection provisions.

4.2.1 Lot formation. A lot shall consist of the propylene glycol dinitrate offered for acceptance at one time which has been produced by one manufacturer, at one plant, during a period of time not exceeding 20 hours, 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 6.5).

4.2.2 Sampling. Using a dipper made of rubber or an approved plastic, a sample of approximately 50 milliliters (mL) of propylene glycol dinitrate shall be removed from each lot. The sample shall be transferred to a stoppered bottle made of rubber or an approved plastic. The bottle shall be labeled to show the lot number and date of manufacture.

4.2.2.1 After disposal of the propylene glycol dinitrate from the sample bottle, the bottle shall be cleaned as follows:

- a. Rinse thoroughly with acetone.
- b. Flush generously with hot tap water (3 minutes under a direct flow is considered adequate).
- c. Rinse with distilled water and dry.

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 requirement of 3.3.

4.3.2 Determination of nitrogen. Prepare the nitrometer as described by MIL-STD-286B, method 209.3.2. Wash the nitrometer generator using 25 mL of glacial acetic acid for each of two rinses. Expel all air

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from the stopcock and sidearm. Cap the sidearm. Add 2 mL of glacial acetic acid to the generator cup and draw it approximately three quarters of the way into the nitrometer. Weigh accurately to 0.1 milligram (mg) a sample containing 0.70 ± 0.05 gram (g) of PGDN and transfer quantitatively to the generator cup. Draw the acid-sample mixture into the generator. Add a 2 mL portion of glacial acetic acid to the generator cup and draw the acid into the generator. Repeat the steps in the previous sentence using a second portion of acid. Add 25 mL of 94.5% sulfuric acid to the generator cup and draw the acid into the generator.

CAUTION: Any temperature rise should be avoided by regulating the rate of sulfuric acid addition. There is a color change from red to purple to yellow red during the addition of the acid and the subsequent steps of the procedure.

Close the top stopcock and immediately open the bottom stopcock. React the sample by gently shaking the top of the generator bulb holding the bottom end of the bulb as the axis.

CAUTION: The lower stopcock must be open until the gas has been generated and any temperature rise in the sample during the reaction must be avoided. Safety shields must be used.

Allow the generator to stand until the mercury settles. Drain the excess mercury until about 50 mm of mercury remain in the bottom of the generator bulb. Close the bottom stopcock, shake the generator bulb vigorously for 3 to 5 minutes, and allow the generator to stand for an additional 5 minutes. Vent any air from the reading tube, the stopcock, and the sidearm of the reading tube. Connect the generator to the reading tube with an airtight connection. Transfer the gas to the reading tube and adjust the mercury level in the compensatory tube to the calibration mark. Then adjust the mercury in the reading tube so that it is level with the calibration mark. Allow the system to come to equilibrium at ambient temperature, make a final adjustment of the mercury level, and read the gas volume.

Calculation:

$$\text{Percent nitrogen} = \frac{A}{W}$$

Where: A = reading of the mercury level on the measuring tube
W = weight of sample (on the moisture-free basis), g

4.3.3 Determination of acidity and alkalinity. Place approximately 50 mL of distilled water into each of two 125-mL Erlenmeyer flasks and add ten drops of bromothymol blue indicator to each flask. Neutralize the contents of the flasks with either 0.01N acid or base to bring the

water to a neutralization point (yellow pH 6.0 - blue pH 7.6). A pale green color indicates the desired neutralization point. Weigh approximately 20 g of raw PGDN from a weighing bottle into one of the flasks. Allow the sample to enter along the inner sides of the flask. Record weight of the sample to the nearest 0.1 mg. Gently mix the PGDN and water with a swirling action. Note any change in color due to the indicator change. Compare the flask containing the PGDN with the blank flask. If the PGDN flask is blue, titrate with standard acid to the neutral point. If the PGDN flask is yellow, titrate with standard base. Use the color of the blank flask to compare the color for the end point. The desired end point is a pale green color.

Calculation:

$$\text{Percent acidity as H}_2\text{SO}_4 = \frac{(\text{mL base} \times \text{normality of base}) \times 4.9}{\text{Sample weight, g}}$$

$$\text{Percent alkalinity as Na}_2\text{CO}_3 = \frac{(\text{mL acid} \times \text{normality of acid}) \times 5.3}{\text{Sample weight, g}}$$

4.3.4 Determination of moisture content. 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 dinitrate (5 to 15 g) to the titration flask. Titrate the sample to a potentiometric end-point.

Calculation:

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

where:

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

4.3.5 Determination of stability. Filter a portion of the propylene glycol dinitrate sample through two thicknesses "S. and S." Number 604 filter paper or equivalent. Transfer a 2 mL portion of the filtered material by means of a pipette, to each of three test tubes which are 140 mm long, 13 mm internal diameter and 16 mm outside diameter. Care should be taken during the transfer not to leave droplets of the propylene glycol dinitrate on the sides of the test tubes. Stopper each tube by means of a new, tightly fitting cork through which passes a tightly fitted glass rod equipped with a platinum holder for a strip of standard potassium iodide starch-indicator paper. Using forceps, hang a strip of standard potassium iodide starch-indicator paper on the platinum holder. The standard potassium iodide starch-indicator test paper shall normally be approximately 25 mm long and 9.5 mm wide. Moisten a horizontal section in the upper half of the standard test paper with a 50 percent by volume solution of pure glycerin in distilled water. This is

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conveniently accomplished by dipping a small diameter glass rod into the glycerin-water solution, and as the rod is withdrawn, making contact with the side of the container so as to minimize the volume of the solution adhering to the rod. Draw the rod across the paper strip so as to produce a level and distinct line of demarcation on the lower edge of the wet area. Prepare a blank by suspending a moistened strip of the standard test paper in a clean dry tube. Adjust the temperature of the heat tube bath to $82.2^{\circ} \pm 1^{\circ}\text{C}$ and insert all four test tubes into the heating solution. The depth of immersion of the test tubes into the heating solution should be approximately 50 mm. The bath should be placed in such a position that the test tubes are viewed against a white background illuminated by bright diffused daylight. Note the time of insertion of the tubes into the bath. During the test, the line of demarcation on the standard test paper should be 76 mm above the level of the propylene glycol dinitrate in the test tube. The line of demarcation in the blank tube is regulated at an equivalent height in the tube. Consider the end-point of the test to be the first appearance of any discoloration at the line of demarcation between the wet and the dry portion of the test paper in the sample tube which is in excess of the discoloration observed at the same position on the test paper in the blank. Note the time for completion of the test to the nearest minute. Consider the minimum time for the three tubes to represent the heat test value of the sample. After the test, rinse all rods and tubes with acetone to remove the propylene glycol dinitrate. Wash with warm soapy water, rinse thoroughly with tap water, then with distilled water and dry in a steam oven at approximately 90°C .

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

5. PACKAGING

5.1 Packing.

5.1.1 Level C. Propylene glycol dinitrate is classed as a "Forbidden Explosive" by Section 49 of the Code of Federal Regulations (CFR). For shipment by public highways, the appropriate section of Section 49 of the CFR shall be followed.

5.2 Marking. The marking of interior packages and exterior shipping containers shall be in accordance with instructions contained in Section 49 of the CFR and in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. Propylene glycol dinitrate is intended for use as a propellant ingredient.

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 a certified analysis and/or a test report is required

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

<u>Data Requirement</u>	<u>Applicable DID*</u>
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 Safety precautions. The safety precaution requirements of the "Contractor's Safety Manual for Ammunition, Explosives, and Related Dangerous Material" (DOD 4145.26M) are applicable and should be specified in the contract or order as required by the Armed Services Procurement Regulations (ASPR) 1-323.

NOTE: When this specification is used as part of the description of work to be accomplished by a Government activity, the safety precaution requirements of "Ammunition and Explosives Ashore" (OP 5) should be made applicable.

6.4 Toxic effects. Toxic effects may occur from the inhalation of vapors from propylene glycol dinitrate or the absorption from direct contact with the skin.

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

Custodian:
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Navy - OS

Project Number:
9135-N089

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