MILITARY SPECIFICATION

PROPELLANT, uns-DIMETHYLHYDRAZINE

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

1. SCOPE

1.1 This specification covers the requirements for unsymmetrical dimethylhydrazine (CH_3)₂ NNH_2 propellant (UDMH).

2. APPLICABLE DOCUMENTS

2 1 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.

SPECIFICATIONS

Military

MIL-P-27401	Propellant Pressurizing Agent, Nitrogen
MIL-P-27407	Propellant Pressurizing Agent, Helium

STANDARDS

Military

MIL-STD-129	Storage
MIL-STD-172	Color Code for Containers of Liquid Propellants

FSC 9135

(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.)

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 Publications

ASTM Standards, Parts 17, 18, and 30

(Copies of ASTM publications may be obtained upon application to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

Department of Transportation

49 CFR 170-190

Department of Transportation Hazardous Materials Regulations

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.)

Manufacturing Chemists' Association, Inc.

MCA Manual L-1

A Guide for the Preparation of Warning Labels for Hazardous Chemicals (1961).

(Application for copies should be addressed to the Manufacturing Chemists' Association, Inc., 1825 Connecticut Avenue, N.W., Washington, D.C. 20009).

2.2.1 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 Chemical and physical properties. The chemical and physical properties of the propellant shall conform to those listed in table I when tested in accordance with the applicable test methods.

Table	τ.	Chemical	and	Physical	Properties
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Properties	Limits	Test Paragraph
uns-Dimethylhydrazine assay (percent by weight)	98 min	4.5.2
Water (percent by weight)	0.3 max	4.5.2
Particulate (milligrams per liter)	10 max	4.5.3

- 3.2 Limiting values. The following applies to all specified limits in this specification: For purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limitation value, in accordance with the rounding-off method of the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E29).
- 3.3 Filter. A filter with a 10-micron nominal and 40 absolute rating shall be installed between the manufacturer's plant system and the container to be filled for delivery.
- 3.4 Qualitative. The propellant shall be a homogeneous liquid when examined visually by transmitted light.

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 in the contract or order, the supplier 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 prescribed requirements.

- 4.2 Classification of tests. The inspection and testing of the propellant shall be classified as quality conformance tests.
- 4.3 Test conditions. The test conditions are described under the individual tests to which they apply.
- 4.4 Quality conformance tests. Quality conformance tests shall consist of:
 - (a) Individual test 4.4.1
 - (b) Sampling tests 4.4.2
- 4.4.1 Individual test. The propellant shall be subject to the following test as described under 4.5.
 - Examination of product 4.5.1
- 4.4.2 Sampling tests. The propellant shall be selected in accordance with 4.4.2.1 and subjected to the following tests as described under 4.5.
 - (a) uns-Dimethylhydrazine assay 4.5.2
 - (b) Water 4.5.2
 - (c) Particulate 4.5.3
 - 4.4.2.1 Sampling plan.
 - 4.4.2.1.1 Lot. A lot shall consist of one of the following:
- (a) The propellant produced in not more than 24 consecutive hours from a continuous process which is used to fill shipping containers directly from the process output. A continuous process shall be the production of product by continuous input of raw materials and output of finished product by one manufacturer in one plant with no change in manufacturing conditions or materials.

- (b) The propellant produced from individual runs of a batch process which is used to fill shipping containers directly from the process output. A batch process shall be the production of product from single additions of raw materials which are reacted and purified forming the product.
- (c) The propellant from either or both the continuous and batch processes which is held in a single storage tank and subsequently withdrawn to fill shipping containers. The product shall be homogeneous at the time of withdrawal and shall not be added to while being withdrawn. After each addition to the storage tank, the contents shall constitute a separate lot.
- 4.4.2.1.2 Sample. A sample consists of not less than 600 milliliters (ml) of propellant. Unless otherwise specified, quality conformance tests shall be made on the sample of the propellant taken directly from the shipping containers. When required, the sample shall be forwarded to a laboratory designated by the procuring activity for subjection to the quality conformance specified herein. The bottles intended for sampling shall be specially cleaned and handled in accordance with procedure described in 4.5.3.
- 4.4.2.1.3 Drums. The number of drums selected for sampling from each lot shall be in accordance with table II. The propellant from each container sampled shall constitute a separate sample.

Table II. Sampling for Test

Number of containers in lot	Number of containers to be sampled
2-25	2
26-150	3
151-1,200	5
1,201-7,000	8

4.4.2.1.4 Portable tanks, cargo tanks, and tank cars. Each portable tank, cargo tank, or tank car shall constitute a lot. Unless

otherwise specified, the sample shall be composited into one sample when one-third portions are withdrawn from the bottom, center, and top thirds of the tank.

- 4.4.2.1.5 Other containers. Unless otherwise specified, other containers of 100 gallons or less water capacity shall be sampled in accordance with 4.4.2.1.3 and other containers greater than 100 gallons water capacity shall be sampled in accordance with 4.4.2.1.4.
- 4.4.3 Rejection. When any sample of the propellant tested in accordance with 4.5 fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected.

4.5 Test methods.

- 4.5.1 Examination of product. The propellant shall be visually examined while performing test specified in 4.5.3 to determine requirements as specified herein. Examination to ensure that the material conforms to paragraph 3.4 shall be conducted after the sample has been transferred to the 500-ml graduated cylinder.
- 4.5.2 uns-Dimethylhydrazine assay and water. The propellant and water content of the sample shall be determined by the following method.

4.5.2.1 Gas chromatographic method.

- 4.5.2.1.1 Suggested column preparation. Weigh 10 grams of polyethylene glycol 400 and 20 grams of Anakrom B support material into separate beakers. Dissolve the polyethylene glycol 400 in reagent grade methylene chloride (dichloromethane). The final volume of the solution should be approximately that of the support material. Pour the support material into the polyethylene glycol 400 (stationary phase) solution with gentle stirring. Evaporate the solvent by spreading the mixture in a tray. Occasionally turn the mixture gently during the drying process. The column packing material is dry when it becomes a freeflowing powder. Fill a 1/4 inch x 1 meter (39.4 inches) stainless steel tube by pouring the prepared material through a small funnel at-The bottom of the tubing is plugged with a small tached to one end. wad of glass wool. Tap gently or use a mechanical vibrator to facilitate packing. When the column is full, plug the inlet with glass wool and bend the tubing to the configuration required by the chromatograph oven.
- 4.5.2.1.2 Procedure. Install the prepared column into the gas chromatograph but do not connect the column to the detector inlet.

Condition the column for at least 4 hours by heating at 302°F (150°C) with the carrier gas (helium) flow set at approximately 50 milliliters per minute. After conditioning the column, set the column oven temperature at 230°F (110°C) and connect the column to the detector inlet. Adjust the carrier gas flow to 80-120 milliliters per minute. If the gas chromatograph is equipped with separate injector and detector temperature controls, set the detector at 302°F (150°C) and the injector at 230°F (110°C). Saturate the column by injecting two or three 10-microliter samples of propellant. Saturate the column prior to each series of analyses. When more than 30 minutes elapse between the elution of the hydrazine and the injection of a new sample, resaturate the column with one 10-microliter injection of propellant sample. Use a clean, dry 10-microliter hypodermic syringe and draw up 8 microliters of sample. Invert the syringe and expel the gas bubbles. Carefully set the syringe plunger to the 3-microliter mark and wipe the tip with a piece of tissue without touching the open end of the needle. Quickly inject the sample into the instrument injection port and then withdraw the syringe immediately. Measure the areas of all peaks in the chromatogram. The analyst may vary the temperature, flow rate, column size, and sample size to optimize the analysis.

4.5.2.1.3 Calculations.

$$\%UDMH = \frac{A}{\Sigma A_{i}} \times 100$$

$$\%H_{2}O = \frac{A}{\Sigma A_{i}} \times 100$$

Where:

AUDMH = the measured area of the UDMH peak multiplied by its signal attenuation factor.

 $^{A}_{H_{2}O}$ = the measured area of the $_{2}O$ peak multiplied by its signal attenuation factor.

 ΣA_i = the sum of all the measured peak areas in the chromatogram multiplied by their respective signal attenuation factors.

Assumption: The thermal conductivities of all components in the sample are equal.

4.5.2.1.4 Equipment and reagents. The following equipment and reagents shall apply as test conditions of 4.5.2.

(a) Equipment

- (1) Gas chromatograph: incorporating a thermal conductivity detector.
- (2) Recorder: potentiometric strip chart, 0-1 millivolt, 1 second F.S. response, with integrator.
- (3) Tubing: stainless steel, 1/4 inch 0.D. x 1 meter (39.4 inches).
- (4) Hypodermic syringe: 10 microliter, fixed needle.
- (5) Regulator: helium, to fit the cylinder.

(b) Reagents

- (1) Anakrom B: 90/100 mesh, Analabs, Inc., 9 Hobson Ave., Hamden, Conn. 06518, or equivalent.
- (2) Polyethylene glycol 400; Carbowax 400; or equivalent.
- (3) Methylene chloride: ACS reagent grade.
- (4) Helium gas: conforming to MIL-P-27407.
- 4.5.3. Particulate. The propellant sample shall be tested for contamination in accordance with ASTM Designation D-2276-65T, Method A, with the following exceptions.
- 4.5.3.1 Mix the sample thoroughly by shaking the sample container. Immediately pour 500 ml of the sample into a clean 500-ml graduated cylinder. Use this 500 ml of propellant for the particulate analysis.
- 4.5.3.2 Use a solvent resistant filter disc made from such materials as Millipore LSWP 04700, (Mitex-Teflon), Millipore URWP 04700,

(Solvinert), or Gelman VF-6 (Fluoride-Metricel), plain, white, 10 ± 3 microns, 47 mm diameter instead of the filter specified in Method 2276-65T.

- 4.5.3.3 The drying oven temperature shall be 158°F (70°C) instead of the 194°F (90°C) specified in Method 2276-65T.
- 4.5.3.4 Filtered isopropyl alcohol shall be used for rinsing the sample bottle and filter holder instead of petroleum ether specified in Method 2276-65T.
- 4.6 Preparation for delivery inspection. The preservation, packaging, packing, and marking for shipment and storage of the propellant shall be inspected to determine compliance with the requirements of section 5 of this specification.

5. PREPARATION FOR DELIVERY

- 5.1 Packaging. The propellant shall be packaged in containers as specified by the procuring activity. All packaging shall comply with the requirements of the Department of Transportation (DOT) Regulations 49 CFR 170-190, or DOT special permit in accordance with 49 CFR 170.13 obtained by the shipper in conjunction with the Commander, Headquarters Military Traffic Management and Terminal Service, Attn: Safety Division (TES), Washington, D. C. 20315.
- 5.1.1 Approved drums. Drums which are approved by DOT for this propellant shall conform to DOT specifications 5, 5A, 5C, or 17E (singletrip) of Types 304 or 347 stainless steel; 17C (singletrip) of carbon steel; or 42B of aluminum with fixed heads only and bungs not over 2.3 inches diameter.
- 5.1.2 Approved cargo tanks. Cargo tanks which are approved by DOT for this propellant shall conform to DOT specifications MC 300, MC 301, MC 302, MC 303, MC 304, MC 305, MC 306, MC 307, MC 310, MC 311, or MC 312 of carbon steel or stainless steel with no bottom outlets and equipped with approved steel safety valves.
- 5.1.3 Approved tank cars. Tank cars which are approved by DOT for this propellant shall conform to DOT specifications 103W, 103C-W, 105A100-W, 111A100-W-4 of carbon steel or stainless steel with approved steel safety valves. 103W tank cars shall not be equipped with bottom outlets. Tank cars conforming to specifications 105A200-W, 105A300-W, 105A400-W, 105A500-W, 105A600-W shall be restenciled 105A100-W and be equipped with safety valves of the type and size used on 105A100-W tank cars.

- 5.1.4 Other containers. Other containers of any type which do not presently have DOT approval shall conform to the requirements of DOT special permit.
- 5.2 Preparation of containers. Prior to filling, the contractor shall establish the condition of all containers to insure that they are free from contamination and suitable for shipment and storage. Contractor owned containers shall be cleaned and repaired by the contractor at his own cost. Leased or government owned containers shall be cleaned and repaired in accordance with the schedule established in the contract or purchase order.
- 5.2.1 Cleaning and repair. Unless otherwise provided for in the contract or purchase order, all containers shall be visually inspected internally and externally for the presence of water, rust, scale, oil film, or other foreign matter, and physical damage. Any physical damage which would endanger safe transportation of the propellant shall be repaired prior to reuse. If evidence of internal contamination is found, the containers shall be recleaned by a suitable method to remove the contamination. Internal inspections on cargo tanks or tank cars used in exclusive continuous service need be made only upon initial entry into that service or at any required retest or overhaul.
- 5.2.2 Gaskets. Gaskets used to seal container openings shall be polytetrafluoroethylene or other material compatible with the propellant and approved for use by the procuring activity. The contractor shall assure that all gaskets are serviceable and furnish new gaskets when necessary so that a tight seal is assured.
- 5.3 Filling. Containers shall not be entirely filled. Sufficient space shall be left in each container to assure that no leakage or distortion of the container occurs as specified by DOT requirements. After filling of containers, the space above the liquid level shall be filled with contractor furnished nitrogen conforming to MIL-P-27401. Type I, at not less than atmospheric pressure.
- 5.4 Labeling and marking. Each container shall be labeled and placarded in accordance with MIL-STD-129 and established DOT requirements or DOT special permit. In addition, an identification tag, precautionary label, and container color code shall be used.
- 5.4.1 Identification tag. Unless otherwise specified in the contract or purchase order, an identification tag impervious to climatic conditions shall be wired to the outlet port of each container

and shall contain the following information: Propellant name, specification number with revision letter, type number (if applicable), FSN number, quantity, name of manufacturer, name of contractor (if different from manufacturer), and date of manufacture.

- 5.4.2 Precautionary label. A precautionary label prepared in accordance with MCA Manual L-1 shall be applied to each drum (6.6).
- 5.4.3 Container color code. Each drum shall be color coded in accordance with MIL-STD-172. The exact name identification to be marked on the outside of the container shall be "uns-Dimethylhydrazine". Any other name identification shall be obliterated by removing or overpainting.

6. NOTES

- 6.1 Intended use. The propellant covered by this specification is intended for use as a fuel in rocket engines.
 - 6.2 Definition.
- 6.2.1 Particulate. The undissolved solids retained on a 10-micron filter membrane.
- 6.3 Density. For information purposes, the range of density for material conforming to this specification is 0.783-0.786 gm/ml at $77^{\circ}F$ (25°C).
- 6.4 Ordering data. Procurement documents should specify the following:
 - (a) Title, number, and date of this specification.
 - (b) Method of shipment, type, and capacity of containers.
 - (c) Quantity by weight in pounds (avoirdupois).
- (d) That two copies of the test report, signed by the contractor's representative, listing values obtained on all tests (qualitative where method provides), should accompany each shipment delivered to the consignee. In addition, one copy should be furnished the AFRPL (RPORS), Edwards, California 93523.

- 6.5 Highway safety. To promote safety in the transportation of propellants in interstate commerce by motor vehicle, the shipper should assure that each driver possesses an MCA Chem-Card-Transportation Emergency Guide No. CC8. A complete manual of cards of the individual cards are available from the Manufacturing Chemists' Association, 1825 Connecticut Avenue, N.W., Washington, D. C. 20315.
- 6.6 Precautionary labels. Precautionary labels are prepared in accordance with MCA Manual L-1. For those propellants which do not have specifically prescribed labels, the principles for the preparation of the labels are used. There does not have to be exact agreement between labels from different sources as long as the intent of the manual is complied with.
- 6.7 Pollution control. U. S. Public Laws dictate increased effort to improve air, land, and water pollution control of toxic propellant vapors, leaks, spills, and disposal during all phases of manufacture, transfer, storage, and transportation operations. The manufacturer/supplier is enjoined to approach the appropriate pollution control district to mutually resolve all problem areas, and to develop adequate control and disposal methods for situations which are likely to develop in any of the phases.

Custodians: Preparing Activity:

Army - MI Air Force - 12

Navy - AS

Air Force - 12

Review Activities: Civilian Agency Interest:

Air Force - 19, 68 NAS

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