MIL-STD-1775
4 Jun 82

MILITARY STANDARD

# PROPELLANT, HYDRAZINE-UNS-DIMETHYLHYDRAZINE 50/50 BLEND

FSC 9135

DEPARTMENT OF DEFENSE
Washington, DC 20360

Establishes Use Limits for the Rocket Propellant Hydrazine/Uns-dimethylhydrazine, 50/50 Blend.

MIL-STD-1775

1. This Military Standard is approved for use by all Departments and Agencies of the Department of Defense.

2. Recommended corrections, additions or deletions should be addressed to: Director, The Air Force Rocket Propulsion Laboratory, Attn: LKCP, Edwards AFB CA 93523.

# FOREWORD

This military standard was prepared for use by governmental agencies involved with the use of hydrazine-unsymetrical dimethylhydrazine (50/50) mixture, procured in accordance with MIL-P-27402, for the Titan III propulsion system. This military standard covers the on-site use limits and test procedures for this propellant. the use limits represent the minimum purity level acceptable for operational use and allows for some quality degradation from the requirements of MIL-P-27402 due to handling the propellant after procurement by the Air Force.

CONTENTS

## 1.0 SCOPE

1.1 Principal Statement. This military standard covers the on-site use limits and analytical test procedures for propellant hydrazine-unsymmetrical dimethylhydrazine, a 50/50 mixture of hydrazine ($N_2O_4$) and unsymmetrical dimethylhydrazine (UDMH). This propellant is procured in accordance with MIL-P-27402 and utilized in the Titan III propulsion system.

## 2.0 REFERENCED 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 standard to the extend specified herein.

SPECIFICATIONS

| | |
|---|---|
| MIL-P-25604 | Propellant, uns-Dimethylhydrazine |
| MIL-P-26536 | Propellant, Hydrazine |
| MIL-P-27402 | Propellant, Hydrazine-uns-Dimethylhydrazine (50% $N_2O_4$ - 50% UDMH) |
| MIL-P-27407 | Propellant Pressurizing Agent, Helium |

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

2.2 Other Publications. The following documents form a part of this standard to the extend specified herein. Unless otherwise indicated, the issue in effect on the date of invitation for bids or request for proposal shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

| | |
|---|---|
| E 29 | Recommended Practices for Designating Significant Places in Specified Limiting Values |
| D 2276 | Particulate Contaminant in Aviation Turbine Fuels |

(Copies of ASTM Publications may be obtained upon application tot he American Society for Testing and Materials, 1916 Race Street, Philadelphis PA 19103.)

## 3.0 DEFINITIONS

3.1 Single Phase Liquid. A single phase liquid is devoid of any visible foreign liquid, but may contain solid material as permitted within this standard.

3.2 Use Limits. Use limits are a level of quality which allows for degradation of the propellant after procurement due to handling, shipping and transfers. Test data exceeding one or more of these limits indicates the propellant is unsuitable for the use intended.

## 4.0 GENERAL REQUIREMENTS

Not applicable

## 5.0 DETAILED REQUIREMENTS

5.1 Use Limits. The chemical composition of the propellant for operational purposed shall conform to the requirements specified in Table I.

TABLE I
Use Limits

| Constituent | Limit | Test Paragraph |
|---|---|---|
| Hydrazine + UDMH, percent by weight 2/ | 98.0 min. | 5.5.2 |
| UDMH, percent by weight 2/ | 46.3 min | 5.5.2 |
| Hydrazine, percent by weight | 51.0 $\pm$ 0.9 | 5.5.2 |
| Formaldehyde Dimethylhydrazine percent by weight | 1.5 max | 5.5.2 |
| Water, percent by weight 1/ | 2.0 max | 5.5.2 |
| Particulate, mg/L | 25 max | 5.5.3 |

NOTES:

1/ This limit indicates excessive atmospheric contamination.

2/ Percentage includes Amines and FDMH plus any other impurities not listed in this table.

5.2 Limiting Values. The following applies to all specified limits in this standard. 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: E 29).

5.3 Qualitative. The propellant shall be a single phase liquid (3.1) when examined by transmitted light (5.5.1).

5.4. Sampling. Sampling is required in order to obtain a small representative portion of a large quantity of material, and it is one of the most important operations in the analysis of a propellant. A propellant sample shall consist of not less than 600 milliliters (ml) of propellant.

5.4.1 Precautions in Sampling. The operator taking the sample will use extreme care and good judgment to assure that the sample is truly representative of the product being sampled. Compliance with local safety regulations for handling hydrazine fuels is mandatory. The following cautions apply:

a. Use of proper safety equipment.

b. Assure gloves are clean.

c. Assure sampling equipment and sampling containers are cleam.

d. Assure proper sampling equipment is being used.

e. Agitate the contents of the container to be sampled, if possible, to assure that it is properly mixed.

f. Assure precautions are taken to avoid contamination of the sample or storage containers.

5.4.2 Sampling Bulk Containers. The following procedures shall apply to propellant containers with a fluid capacity greater than 55 gallons.

5.4.2.1 Sampler. A sampler consisting of a cylinder, Hoke Part No. 8HD1000 or equivalent, and two valves, Hoke Part No. 2462-L84Y or equivalent, shall be used.

5.4.2.2 Sampling Procedures. The propellant container shall be sampled as follows:

a. Assure the sampler has been cleaned and tagged in accordance with 5.4.2.3.

b. After removing the protective caps, connect the sampler into the sampling line of the container (see Figure 1).

c. Open the sample line purge valve.

d. Open the sampling valve and purge the sample line with a quantity of propellant equal to or greater than the volume of sample line.

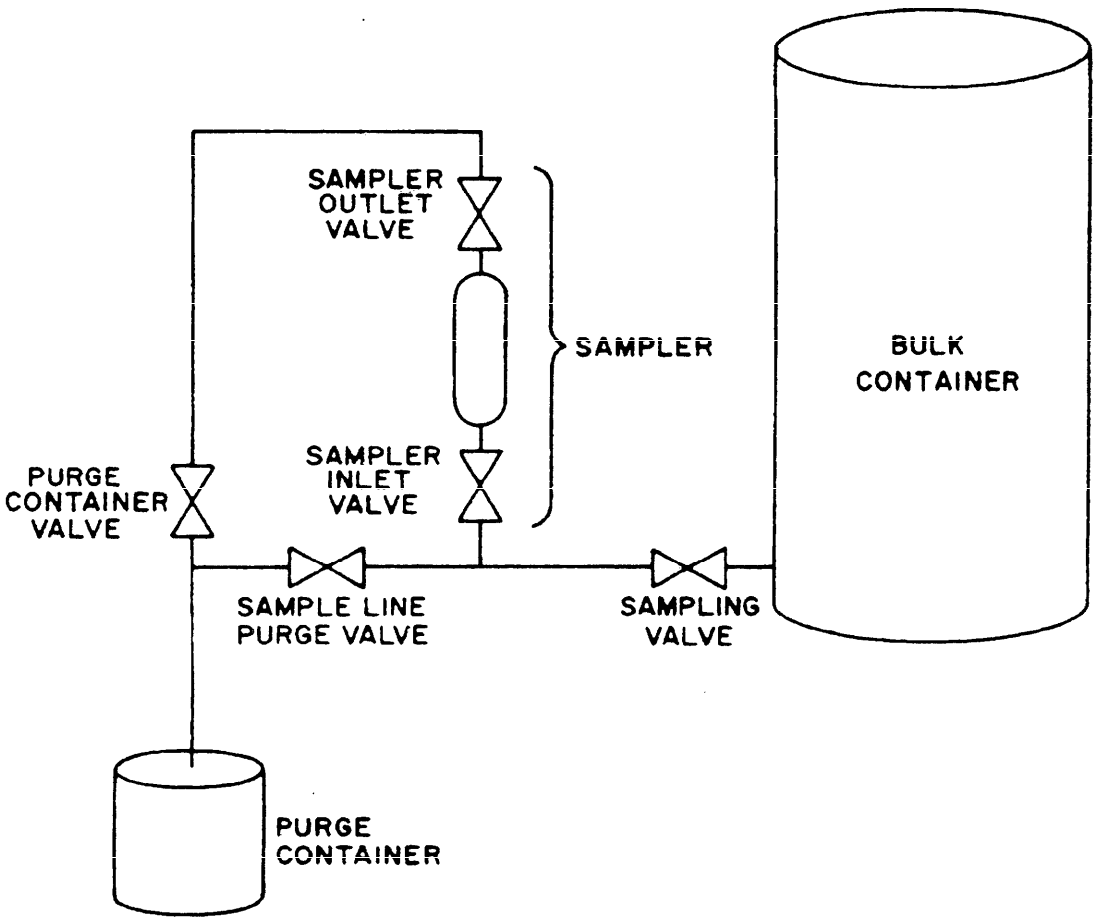


FIGURE 1. Sampling configuration

e. Close sample line purge valve and open sampler inlet and outlet valves, and the purge container valve.

f. When propellant starts flowing into the purge container, close the sampler outlet, sampler inlet, and sampling valves.

g. Disconnect the sampler at the sampler outlet valve.

h. To allow for ullage, predetermine 10 percent of the sampler volume. Open the sampler inlet and outlet valves, and the sample line purge valve to discard this quantity of propellant into the purge container. Close the sampler inlet and outlet valves and disconnect the apparatus and sampler from the bulk container.

i. Replace all protective caps. Wash all parts with water.

j. Prepare sample identification tag in accordance with 5.4.4.

k. Submit sample to laboratory for analysis.

l. Properly dispose of propellant in purge container.

m. Reconnect purge container to sampling configuration.

5.4.2.3. Sampler Cleaning Procedures. When sampling equipment and containers have been certified clean to the level required by TO 42C-1-11 for the propellant to be sampled by Air Force or contract cleaning facilities, no other cleaning is required. The following instructions are provided where a cleaning facility is not available.

a. Drain any remaining liquid from the sampler.

b. Remove propellant vapors from the sampler by purging with nitrogen.

c. Add distilled water, which has been filtered through a 10-micron filter, to the sampler. Shake the sampler and drain the fluid.

d. Add isopropyl alcohol, which has been filtered through a 10-micron filter, to the sampler. Shake the sampler and drain the fluid. Then purge the sampler with dry filtered nitrogen.

e. Place the sampler in a vertical position in an oven (with sampler valves open) at 120°F to 130°F for four hours.

f. Purge sampler again with dry filtered nitrogen.

g. Pressurize sampler (100 psig) with dry filtered nitrogen and test for leakage. Correct as required.

h. Release pressure to between 5-20 psig. Assure valves are in a closed position.

i. Place sampler in polyethylene bag. Seal bag and attach certification that sampler was cleaned in accordance with paragraph 5.4.2.3 of this military standard.

5.4.3 Sampling Drums. The following procedures shall apply to propellant containers with a fluid capacity of 55 gallons or less.

5.4.3.1 Sampling Equipment. The following equipment, or its equivalent, shall be used for sampling drums.

a. Screw cap bottle, Sargeant-Welch Co. Part No. S-8240D or E.

b. Screw cap, Sargeant-Welch Co. Part No. S-9611.

c. Conical cap liners of polyethylene, teflon, or Kel-F only will be used.

d. Acid pump, Siphon Type Polyethylene part No. S-526 Sargeant-Welch.

e. Acid pump, Carboy, Polyethylene Hand Operated part No. S-548-10 Sargeant-Welch.

f. Purge container.

5.4.3.2 Sampling Procedure. The propellant container shall be sampled as follows:

a. Open the container sampling port and insert pump.

b. Start flow of propellant from pump and purge with about 1 pint to 1 quart of propellant. Stop flow of propellant.

c. Place clean sampling bottle (5.4.3.3) over nozzle of pump and start flow propellant.

d. Fill bottle 1/8 - 1/4 full and stop flow of propellant.

e. Cap and shake bottle.

f. Discard this propellant

g. Replace bottle over nozzle and start flow of propellant.

h. After the desired volume has been obtained, stop the flow, cap the sample bottle, remove the pump, and close container sampling port. NOTE: Do not fill sample bottles over 90% full.

i. Rinse sampling equipment and sample bottle with distilled water.

j. Prepare sample identification tag in accordance with 5.4.4.

5.4.3.3 Sample Bottle and Cap Cleaning Procedures. Sample bottles shall be cleaned as follows:

a. Dispose of any remaining liquid.

b. Rinse in warm tap water in a hood.

c. Wash in hot soap and water.

d. Rinse in warm tap water.

e. Rinse with denatured alcohol and purge with dry filtered nitrogen.

f. Place inverted sample bottle in oven at 180°F - 200°F for four hours, and then purge again with dry filtered nitrogen.

g. Place sample bottle and cap in polyethylene bag. Seal bag and attach certification that sample bottle was cleaned in accordance with paragraph 5.4.3.3 of this military standard.

5.4.4.4 Tagging of Samples. Each sample will be identified by means of a tag bearing the following information.

a. Name, location, and telephone number of activity submitting the sample.

b. Sample number. The sample number is the submitting activity identifier and should be numbered consecutively during a calendar year, i.e., 78-1, 78-2, and 78-3 would be the first three samples of 1978. Assign the same number to all sample containers that are taken at the same time from the same tank or other container.

c. Name of product.

d. Number of this military standard.

e. Batch/Lot Number if the sample was taken from delivery trailer or drum.

f. Tank, trailer, or car number if transporter is sampled

g. Quantity sample represents.

h. Date sampled.

i. Type of tests required, periodic, resample, preload, etc

j. If the sample is from a missile, indicate the missile number, complex site, and operational phase.

k. Sampler serial number if applicable.

5.5 Test Methods.

5.5.1 Examination of Product. The propellant shall be visually examined while performing test specified in 5.5.4 to determine compliance with the requirement of paragraph 5.3. This examination shall be conducted after the sample has been transferred to the 500-ml graduated cylinder.

5.5.2 Hydrazine-uns-Dimethylhydrazine Assay, Water, Formaldehyde Dimethylhydrazone (FDMH), and Amines. the propellant, water, and amine content shall be determined by the following method.

5.5.2.1 Column Preparation. Weigh 5 grams of polyethylene glycol 400 and 45 grams of 60/80 mesh Fluoropak 80 into separate beakers. Dissolve the polyethylene glycol 400 in a volume of reagent grade dichloromethane which is approximately one-half the volume of the Fluoropak 80. Pour the Fluoropak 80 into the polethylene glycol 400 solution with gentle stirring. Spread the resulting damp powder in a tray and dry the mixture in a vacuum oven at 100°C and less than 50 mm Hg for at least one hour.

Cap one end of an 1/8 inch O.D. by 6 foot stainless steel tube and fill the tube with the prepared column packing by pouring through a small funnel attached to the other end. Tap or mechanically vibrate the tube to insure uniform packing. When the tube is filled, plug both ends with a small wad of glass wool, bend the column to the configuration required by the column oven, and connect the column to the inlet fitting in the oven. Condition the column with carrier gas flowing and the oven set at 120°C for one hour. After conditioning the column connect the other end to the detector and set the carrier gas flow to approximately 25ml/min. and the column oven to 100°C. The inlet and detector temperatures, if separately heated, shall be set to 100°C and 150°C, respectively. the detector current should be set to a nominal sensitivity value recommended for helium by the instrument manufacturer. The column temperature and carrier gas flow may be adjusted by the analyst to provide adequate component resolution for minimum analysis time.

5.5.2.2 Analysis. Equilibrate the column with propellant by injection of two or more 5 microliter samples into the inlet. if more than 30 minutes elapse between analyses a single 5 microliter injection of propellant should re-equilibrate the column. Inject 1-2 microliters of propellant for analysis and record the areas of all peaks in the chromatogram. Each analysis should require less than 15 minutes for elution of all components. The elution order of possible sample components is as follows: Air, $NH_3$, methylamine, dimethylamine, formaldehyde dimethylhydrazone, UDMH, water, methylhydrazine, and hydrazine.

5.5.2.3 Calculations. The following formulas shall be used to calculate the percent by weight of each component appearing in the chromatogram.

$$\%UDMH = \frac{A_{UDMH}\ K_{UDMH}}{\Sigma A_i\ K_i} \times 100$$

$$\%N_2H_4 = \frac{A_{N_2H_4}}{\Sigma A_i\ K_i} \times 100$$

$$\%H_2O = \frac{A_{H_2O}\ K_{H_2O}}{\Sigma A_i\ K_i} \times 100$$

$$\%FDMH = \frac{A_{FDMH}\ K_{FDMH}}{\Sigma A_i\ K_i}$$

Where:

$K_{UDMH}$, $K_{H_2O}$, $K_{FDMH}$ = the normalization factors for UDMH, $H_2O$, and FDMH

$A_{UDMH}$, $A_{N_2H_4}$, $A_{H_2O}$, $A_{FMDH}$ = the measured areas of the UDMH, $N_2H_4$, $H_2O$, and FDMH peaks multiplied by their signal attenuation factors.

$\Sigma A_i\ K_i$ = the sum of all the measured peak areas in the chromatogram multiplied by their respective signal attenuation factors

Assumptions: The normalization factor for $N_2H_4$ = 1.000. The normalization factors for FDMH and other trace volatile impurities = $K_{UDMH}$.

5.5.2.4 Calibration procedure. Obtain the normalization factors for each component by observing the areas produced by a specially prepared mixture, designated the reference standard. Prepare the standard with freshly distilled components assayed by the gas chromatographic procedure of their respective specifications; for example, $N_2H_4$ MIL-P-26536 and UDMH MIL-P-25604. The composition of the mixture should be approximately 50 percent $N_2H_4$, 48 percent UDMH, and two percent $H_2O$. Weigh each component to 0.1 milligram. The order of addition in the standard preparation shall be $N_2H_4$, $H_2O$, and finally UDMH. Calculate the actual composition as follows:

$$\%UDMH = \frac{W_{UDMH} \times assay_{UDMH}}{\text{total weight}}$$

$$\%N_2H_4 = \frac{W_{N_2H_4} \times \text{assay } N_2H_4}{\text{total weight}}$$

$$\% H_2O = \frac{(W_{H_2O} \times 100) + (W_{N_2H_4} \times \%H_2O) + (W_{UDMH} \times \%H_2O)}{\text{total weight}}$$

Where:

$W_{N_2H_4}$, $W_{UDMH}$, $W_{H_2O}$ = the weight of each component.

$W_{N_2H_4} \times \%H_2O$ = the weight of $N_2H_4$ times the percent $H_2O$ determined in the assay as per MIL-P-26536.

$W_{UDMH} \times \%H_2O$ = the weight of UDMH times the percent $H_2O$ determined in the assay as per MIL-P-25604.

Analyze the referenced standard in accordance with 5.5.2.2. Calculate the normalization factors as follows:

$$K_{UDMH} = \frac{\%UDMH \times A_{N_2H_4}}{\%N_2H_4 \times A_{UDMH}}$$

$$K_{H_2O} = \frac{\%H_2O \times A_{N_2H_4}}{\%N_2H_4 \times A_{H_2O}}$$

Where:

$K_{H_2O}$, $K_{UDMH}$ = the normalization factors for $H_2O$ and UDMH

$A_{N_2H_4}$, $A_{UDMH}$,

$A_{H_2O}$ = the measured areas of the $N_2H_4$, UDMH, and $H_2O$ peaks multiplied by their signal attenuation factors.

5.5.2.5 Equipment and Reagents. The following equipment and reagents shall apply as test conditions of 5.5.2

a. Equipment

1) Gas chromatograph: equipped with thermal conductivity detector.

2) Recorder: potentiometric strip chart, 0-1 millivolt, 1 second F.S. response, with integrator (mechanical or electronic).

3) Tubing: stainless steel, 1/8 inch O.D x 6 feet.

4) Hypodermic syringe: 10 microliter, fixed needle.

5) Regulator: helium, to fit the cylinder.

6) Standard screens: 60 mesh and 80 mesh.

7) Vacuum oven, capable of 100°C.

8) Analytical balance, sensitive to 0.1 mg with a 100g minimum capacity.

b. Reagents

1) Fluoropak 80, Analabs, Inc., 80 Republic Dr., North Haven, CT, 06473 or equivalent.

2) Polyethlene glycol 400; or equivalent.

3) Methylene chloride: ACS reagent grade.

4) Helium gas: conforming to MIL-P-27407.

5.5.3 Particulate. the propellant sample shall be tested for contamination in accordance with ASTM Designation D-2276, Method A, with the following exceptions.

5.5.3.1 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.

5.5.3.2 Use a solvent resistant filter disc made from such material as Millipore LSWP 04700, (Mitex-Teflon), Millipore URWP 04700, (Solvinert), or Gelman VF-6 (Fluoride-Metricel), plain white, 10 $\pm$ 3 microns, 47 mm diameter instead of the filter specified in Method D-2276.

5.5.3.4 Filtered isopropyl alcohol shall be used for rinsing the sample bottle and filter holder instead of petroleum ether specified in Method D-2276.

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