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**MIL-PRF-27404D**

**20 April 2012**

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**SUPERSEDING**

**MIL-PRF-27404C**

**1 October 1997**

## **PERFORMANCE SPECIFICATION**

### **PROPELLANT, MONOMETHYLHYDRAZINE**



Comments, suggestions, or questions on this document should be addressed to AFPA/PTPT, 2430 C Street, Bldg 70, Area B, Wright-Patterson AFB OH 45433-7632 or e-mailed to [AFPET.AFTT@wpafb.af.mil](mailto:AFPET.AFTT@wpafb.af.mil). Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <https://assist.daps.dla.mil>.

AMSC N/A

FSC 9135

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

## 1. SCOPE

1.1 Scope. This specification covers the requirements for monomethylhydrazine ( $\text{N}_2\text{H}_3\text{CH}_3$ ) propellant.

## 2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 or 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3 or 4 of this specification, whether or not they are listed.

### 2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

#### DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-PRF-27401     Propellant Pressurizing Agent, Nitrogen

MIL-PRF-27407     Propellant Pressurizing Agent, Helium

(Copies of these documents are available online at <https://assist.daps.dla.mil/quicksearch/> or from the Standardization Document Order Desk, 700 Robbins Avenue, Bldg 4D, Philadelphia PA 19111-5094.)

2.3 Non-government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

#### ASTM International

ASTM D2276     Standard Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling (DoD Adopted)

ASTM E29     Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications (DoD Adopted)

(Copies of these documents are available online at <http://www.astm.org> or ASTM International, 100 Barr Harbor Drive, West Conshohocken PA 19428-2959).

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2.4 Order of precedence. Unless otherwise noted herein or in the contract, in the event of a conflict between the text of this document and the references cited herein (except for related specification sheets), the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

### 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.

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 digit used in expressing the specification limit according to the rounding-off method of ASTM Practice E29, Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications.

3.3 Filter. A filter with a rating of at least 10 micrometer ( $\mu\text{m}$ ) nominal and 40  $\mu\text{m}$  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 clear and colorless homogeneous liquid when examined visually by transmitted light (a trace yellow/straw to green discoloration is allowed when compared to a sample bottle containing purified water).

**TABLE I. Chemical and physical properties.**

Properties	Limits	Test Paragraph
Monomethylhydrazine (% by wt)	98.3	4.3.2
Water (% by wt)	1.5 max	4.3.2
Non-volatile residue (mg/L)	10 max	4.3.3
Particulate (mg/L)	10 max	4.3.4

### 4. VERIFICATION

4.1 Classification of inspections. The inspections shall be classified as quality conformance inspections.

4.2 Quality conformance inspection. Unless otherwise specified (6.2), each filled shipping container shall be considered a lot and shall be sampled. Each sample shall be subjected to the visual examination described in 4.3.1 for conformance to 3.4 and to the tests specified in Table I.

4.2.1 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 propellant taken directly from the shipping container. When required, the sample shall be forwarded to a laboratory designated by the procuring activity for subjection to the quality conformance tests specified herein. The bottle intended for sampling shall be specially cleaned and handled according to the procedure described in ASTM D2276 Section 10 with the exception listed 4.3.4d.

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4.2.2 Rejection. When any sample of the propellant tested in accordance with 4.3 fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected.

### 4.3 Test methods.

4.3.1 Examination of product. The propellant shall be visually examined while performing test specified in 4.3.4 to determine compliance with the requirement as specified herein. Examination to ensure that the material conforms to 3.4 shall be conducted after the sample has been transferred to the 500 mL graduated cylinder.

4.3.2 Monomethylhydrazine (MMH) assay and water. The propellant and water content of the sample shall be determined by one of the Gas Chromatographic (GC) methods described in Appendix A. In case of a dispute, the packed column method (A.2.1) shall be the referee.

4.3.3 Non-volatile residue (NVR). The NVR of the propellant shall be determined in duplicate by either of the methods described in Appendix B. In case of a dispute, the dropwise method (B.2.1) shall be the referee.

4.3.4 Particulate. The propellant sample shall be tested gravimetrically for contamination in accordance with ASTM D2276, with the following exceptions:

a. 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.

b. Use a Mitex disc polytetrafluoroethylene (PTFE) hydrophobic, 10  $\mu$ m, 0.47 millimeters (mm), white filter or equivalent instead of that specified in ASTM D2276.

c. Set the oven temperature to 70°C (158°F).

d. Filtered American Chemical Society (ACS) grade isopropyl alcohol shall be used for rinsing the sample bottle and filter holder instead of petroleum ether specified in ASTM D2276.

## 5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Service or Defense Agency, or within the Military Service's System Commands. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

## 6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. The propellant described by this specification is intended for use as a fuel in rocket engines.

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6.2 Acquisition requirements. Acquisition documents must specify the following:

- a. Title, number, and date of the specification.
- b. Method of shipment, type and capacity of containers.
- c. Quantity by weight.
- d. When a different sampling plan is required (4.2).
- e. Packaging requirements (5.1).

6.3 Subject term (key word) listing.

Fuel  
Rocket engine

6.4 Material Safety Data Sheets. Contracting officers will identify those activities that require copies of completed Material Safety Data Sheets (MSDS) prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

6.5 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

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## APPENDIX A

### MONOMETHYLHYDRAZINE ASSAY AND WATER

#### A.1 SCOPE

A.1.1 Scope. This Appendix describes two Gas Chromatographic methods for the determination of propellant and water content in the sample. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

#### A.2 METHODS

##### A.2.1 Gas chromatographic method – packed column.

A.2.1.1 Assay column preparation. Cap one end of a 1/8 inch (in) outer diameter (OD) by 6 foot (ft) nickel tube and fill the tube with the Tenax®-TA 60/80 mesh column packing by pouring through a small funnel attached to the other end. Tap or mechanically vibrate the tube to ensure 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 helium carrier gas flowing and the oven set at 200°C for one hour. After conditioning the column, connect the other end to the detector and set the carrier gas flow to approximately 25 milliliters per minute (mL/min), and the column oven to 100°C. The inlet and detector temperatures, if separately heated, shall be set to 150°C and 210°C, respectively. The detector current should be set to a nominal sensitivity value recommended for helium by the instrument manufacturer.

A.2.1.2 Procedure. Equilibrate the column with propellant by injection of three or more 1 microliter (µL) samples into the inlet. If more than 30 minutes elapse between analyses, a single 1 µL injection of propellant should re-equilibrate the column. Inject 1 µL of propellant for analysis and record the areas of all peaks in the chromatogram using the following time/temperature program:

Helium carrier gas flow	45 mL/min
Initial temperature	100 °C
Initial time	2 min
Rate	20 °C/min
Final temperature	200 °C
Final time	3 min

Each analysis should require less than 15 minutes for elution of all components. Hydrazine elutes at the leading edge of the MMH peak. If observed, traces of hydrazine shall be included in the reporting of MMH.

The elution order of possible sample components is as follows:

Air < ammonia < water < methylamine < hydrazine < MMH.

##### A.2.1.3 Calculations.

$$\%MMH = \frac{A_{MMH} \times 100}{\sum A_i}$$

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$$\%H_2O = \frac{A_{H_2O} \times 100}{\sum A_i}$$

where

$A_{MMH}$  = The measured area of the MMH peak multiplied by its signal attenuation factor.

$A_{H_2O}$  = The measured area of the H<sub>2</sub>O peak multiplied by its signal attenuation factor.

$\sum A_i$  = The sum of all of the measured peak areas multiplied by their respective signal attenuation factors.

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

A.2.1.4 Equipment and Reagents. The following equipment and reagents shall apply as test conditions of A.2.

a. Equipment:

(1) Gas chromatograph with a thermal conductivity detector (TCD).

(2) Agilent ChemStation or equivalent.

(3) Packed column may be commercially obtained from Grace Discovery Sciences, 2051 Waukegan Road, Deerfield IL 60015-1899.

b. Reagents:

(1) Tenax®-TA column packing 60/80 mesh.

A.2.2 Gas chromatographic method - capillary column.

A.2.2.1 Procedure. A 30 meters (m) x 0.53 mm, 1.00 µm film thickness Restek Stabilwax-DB capillary column, a TCD, and a split/splitless injector with a glass wool packed split liner shall be used. Table A-1 depicts recommended parameters for initial setup. These parameters may vary depending on GC system and detector brands, and may be adjusted to provide adequate component resolution.

The elution order of possible sample components is as follows:

Air > ammonia > methylamine > unsymmetrical dimethylhydrazine (UDMH) > MMH > water > hydrazine > aniline

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## APPENDIX A

**TABLE A-1. GC Recommended parameters**

PARAMETER	CONDITION
Carrier Gas	He
Carrier Flow	10 mL/min
Sample Volume.	1 $\mu$ L (10:1 split)
Inlet Temp.	150 °C
TCD Detector Temp.	200 °C
Carrier + Aux. Flow	~25 mL/min
Carrier + Aux. + Ref. Flow	~65 mL/min
Total (Splitter Bypass) Flow	~90 mL/min
Purge	On
Oven Max	200 °C
Equilibration Time	2.00 min
Initial Temp. ( $T_0$ )	60 °C
Initial Time ( $t_0$ )	3.00 min
Rate (1)	10 °C/min
Final Temp. ( $T_{f1}$ )	100 °C
Final Time ( $t_{f1}$ )	0.00 min
Rate (2)	20 °C/min
Final Temp. ( $T_{f2}$ )	185 °C
Final Time ( $t_{f2}$ )	3.75 min

Allow the instrument to stabilize, condition the column and perform check runs by processing three consecutive injections of 1 - 2  $\mu$ L of sample. The checks must meet the quality control criteria in A.2.2.2.

A.2.2.2 Quality control. The following performance criteria must be met for fuel purity analysis,

GC replicate analyses (3 consecutive)	RSD <sup>a</sup> $\leq$ 10 %
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a. RSD = Standard Deviation \* 100% / Mean

A.2.2.3 Equipment.

- (1) Gas chromatograph equipped with a thermal conductivity detector (TCD) and a split/splitless injector.
- (2) Agilent ChemStation or equivalent.
- (3) A 30 m x 0.53 mm x 1  $\mu$ m Stabilwax DB capillary column.
- (4) Glass wool packed split liner



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## APPENDIX B

### NON-VOLATILE RESIDUE (NVR)

#### B.1 SCOPE

B.1.1 Scope. This Appendix describes two methods for the determination of non-volatile residue of the propellant. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

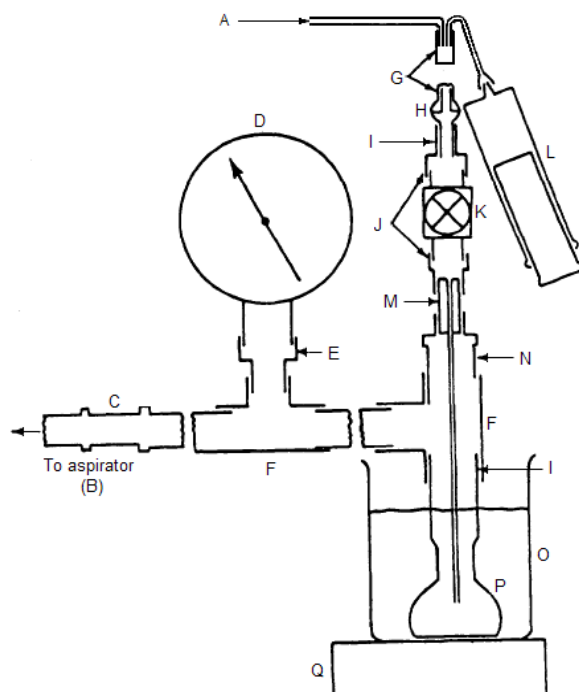
#### B.2 METHODS

##### B.2.1 Dropwise method.

B.2.1.1 Procedure. Clean the unassembled apparatus with detergent and rinse thoroughly with purified water. Dry the parts in an oven at 110°C. Assemble the apparatus as shown in Figure B-1. Fill the water bath with purified water and turn on the heater. Adjust the water level of the bath to approximately one-half inch below the lowest fitting and mark this level on the bath. When the bath temperature reaches boiling, adjust the nitrogen purge to approximately 2 liters per minute (L/min) and turn on the aspirator. With the sample delivery valve closed, the pressure in the apparatus should be below 30 mmHg. Open the sample delivery valve slowly to adjust the apparatus pressure to approximately 60 mmHg. Allow the apparatus to purge for approximately five minutes to remove traces of water. Remove the water bath and dry the outside of the residue vessel with a clean lintless tissue. Disconnect the aspirator from the apparatus and remove the residue vessel using a tissue to hold the vessel. Wipe the outside of the vessel and place it near the analytical balance. After at least twenty minutes, accurately weigh the residue vessel until the weight remains constant to  $\pm 0.1$  mg. Record the weight. Reassemble the apparatus and close the sample delivery valve. Replace the water bath, adjust the water level to the mark, and heat the water to boiling. Transfer 100 mL of propellant to the apparatus. With the nitrogen purge in place, turn on the water aspirator and reduce the apparatus pressure to less than 30 mmHg. Slowly open the sample delivery valve to admit propellant to the residue vessel and adjust the flow such that the apparatus pressure does not exceed 60 mmHg (approximately 2 drops per second). The quantity of propellant in the residue vessel should not exceed 2 - 4 mL at the equilibrium evaporation rate. Adjust the sample delivery valve to maintain the evaporation pressure at less than 60 mmHg. After 100 mL of propellant have been evaporated (approximately 30 min), allow the residue vessel to purge for an additional five minutes to ensure complete evaporation of volatile material. Remove the water bath, disconnect the aspirator, wipe the outside of the residue vessel, and remove it from the apparatus. Allow the residue vessel to stand near the analytical balance for at least twenty minutes before weighing. Weigh the residue vessel as above, and record the weight. To clean the residue vessel reflux 5 mL of 5N ACS grade  $\text{H}_2\text{SO}_4$  (or equivalent) in the vessel for several minutes followed by several rinses of purified water. The vessel may be dried as described above or by gently heating the vessel with a burner while purging with dry nitrogen. Regardless of the drying method used, allow at least twenty minutes for the vessel to equilibrate to ambient temperature and humidity before weighing.

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## APPENDIX B



- |  |   |
|--|---|
| A - GN <sub>2</sub>  | J - Connector (Swagelok SS-400-7-2 or equivalent)                                     |
| B - Water aspirator (glass or polyethylene)                                    | K - Valve (Swagelok SS-ORM2 or equivalent)  |
| C - Connector (polyethylene, separable)  | L - Syringe (100 mL glass with an 8 – 12 in PTFE needle).                             |
| D - Gauge (stainless steel, absolute pressure, 0 - 760 mmHg range)             | M - Rod (PTFE or polyethylene, 1/4 inch OD & a 1/16 OD x 0.025 in wall tube inserted) |
| E - Adapter (Swagelok SS-8-TA-7-4 or equivalent)                               | N - Reducer (Swagelok SS-400-R-8 or equivalent)                                       |
| F - Union Tee (Swagelok SS-810-3 or equivalent; 2 ea.)                         | O - Beaker (glass, 1000 mL capacity)  |
| G - Pipette (100 mL, volumetric, borosilicate glass)                           | P - Flask (25 mL volumetric, borosilicate glass)                                      |
| H - Joints (borosilicate glass, spherical, 12/5, ball, socket and clamp)       | Q - Hotplate (adjustable heat range)  |
| I - PTFE Ferrules (Swagelok T-814-1, T-813-1, T-404-1, T-403-1, or equivalent) |   |

NOTE: All fittings, stainless steel, Swagelok or equivalent.

**FIGURE B-1. NVR apparatus**

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B.2.1.2 Calculation. Calculate the non-volatile residue of the propellant as follows:

$$mg/L\ NVR = \left[ \frac{W_2 - W_1}{V} \right] 1 \times 10^6$$

where,

$W_1$  = Weight of clean vessel in grams (g).

$W_2$  = Weight of residue and vessel in g.

$V$  = Volume of sample used in mL.

Report the averaged result of duplicate NVR determinations.

B.2.1.3 Equipment. The following equipment shall apply as test conditions of B.2.1.

- a. Components described in Figure B-1.
- b. Tubing – polyethylene, ½ inch OD; borosilicate glass, ½ inch OD.
- c. Clamps – hose, to fit ½ inch OD tube.
- d. Five decimal place analytical balance (semi-micro balance).

B.2.2 Hot plate. This method involves the gradual evaporation of a MMH sample on an explosion proof hot plate in a fume hood. The difference in weight before and after evaporation is calculated as NVR.

**WARNING**

Avoid the flammable MMH vapors from coming in contact with any source of ignition by utilizing a certified explosion proof hot plate and a high efficiency fume hood.

B.2.2.1 Procedure. Place three clean borosilicate glass or platinum evaporation dishes in an oven at 110°C for 30 minutes. Label one as a blank and the others as samples 1 and 2. Remove evaporation dishes from the oven and place in a desiccator to cool for one hour. Weigh each clean, dry evaporating dish to the nearest 0.0001 g until the weight remains constant to ± 0.1 mg. Record the sample evaporating dish weight as A and the blank evaporating dish weight as C. Transfer 40 mL of the MMH sample into the evaporating dishes marked 1 and 2. Add 40 mL of water to each sample of MMH in evaporating dishes 1 and 2. Add the same volume of purified water into the blank evaporating dish as was added to the sample dish. Place each evaporating dish on a hot plate which is set to a temperature of 150 - 200°C. Evaporate to dryness, but do not bake. Remove the evaporating dishes from the explosion-proof hot plate and place them in an oven set at 110°C for 30 minutes. Remove dishes from the oven and place in a desiccator for one hour to cool. Weigh each evaporating dish as before to a constant weight while monitoring the room temperature with a narrow range viscosity thermometer (dishes must be weighed at the same temperature). Record the sample evaporating dish weight as B and the blank evaporating weight as D.

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B.2.2.2 Calculations. Calculate the NVR of the propellant as follows:

$$mg/L \text{ NVR} = \left[ \frac{(Wt \text{ } B - Wt \text{ } A) - (Wt \text{ } D - Wt \text{ } C)}{\text{Sample volume, mL}} \right] 1 \times 10^6$$

where,

*A* = Weight of the sample evaporating dish (g).

*B* = Weight of the sample evaporating dish and residue (g).

*C* = Weight of blank evaporating dish (g).

*D* = Weight of blank and water residue (g).

B.2.2.3 Equipment.

- a. Hot plate, explosion proof.
- b. High efficiency fume hood.
- c. Five decimal place analytical balance (semi-micro balance).
- d. Narrow range viscosity thermometer such as an ASTM 17F or equivalent.

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### CONCLUDING MATERIAL

Custodians:

Navy – AS  
Air Force – 68  
DLA – PS

Preparing activity:

Air Force – 68  
(Project 9135-2011-002)

Review activities:

Air Force – 19

Civil Agency:

NASA – NA

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