

**METRIC**

**MIL-PRF-25604F**

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**SUPERSEDING**

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## **PERFORMANCE SPECIFICATION**

### **PROPELLANT, uns-DIMETHYLHYDRAZINE**



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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 uns- dimethylhydrazine (CH<sub>3</sub>)<sub>2</sub>NNH<sub>2</sub> propellant (UDMH).

## 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, standards and handbooks 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-27407 Propellant Pressurizing Agent, Helium

(Copies of these documents are available online at <http://quicksearch.dla.mil>).

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)
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ASTM E29	Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications (DoD adopted)
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(Copies of these documents are available online at <http://www.astm.org> or ASTM International, 100 Barr Harbor Drive, West Conshohocken PA 19428-2959).

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.

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## 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 colorless, homogenous liquid when examined visually by transmitted light.

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

Properties	Limits	Test Paragraph
UDMH (% by weight) <sup>(1)</sup>	98.0 min	4.3.2
Water (% by weight)	0.3 max	4.3.2
Amines <sup>(2)</sup> (Total as DMA & MA % by weight)	1.5 max	4.3.3
NDMA (% by weight) <sup>(3)</sup>	0.01 max	4.3.3
Particulate (mg/L)	10 max	4.3.4
Note: <sup>1</sup> uns-dimethylhydrazine (UDMH) <sup>2</sup> dimethylamine (DMA) and methylamine (MA) <sup>3</sup> N-nitrosodimethylamine (NDMA)		

## 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 glass narrow

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mouth bottles intended for sampling shall be specially cleaned and handled according to the procedure described in ASTM D2276 Section 10 with the exception listed in 4.3.4d of this specification.

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 to ensure that the material conforms to 3.4. The examination shall be conducted after a representative aliquot of the sample has been transferred to a clear glass vessel and compared to the same type vessel containing the same volume of purified water.

4.3.2 Uns-dimethylhydrazine assay, water and amines. The UDMH, water and amine content of the sample shall be determined by the Gas Chromatographic method described in Appendix A.

4.3.3 N-nitrosodimethylamine. The NDMA content of the propellant shall be determined by the method described in Appendix B.

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

a. Mix the sample thoroughly without exposure to air. Immediately pour 500 mL of the sample into a clean 500 mL graduated cylinder. Use this 500mL of propellant for the particulate analysis.

b. Pass the 500 mL sample aliquot through a Mitex disc polytetrafluoroethylene (PTFE) hydrophobic, 10 µm, 0.47 mm diameter, white filter or equivalent.

c. The oven temperature shall be set at 70°C (158°F).

d. Filtered American Chemical Society (ACS) reagent grade isopropyl alcohol shall be used for rinsing the sample bottle and filter holder instead of petroleum ether specified in ASTM D2276. Use a suitable solvent resistant filter disc with an absolute rating of at least 1.0 µm. Wet the filter with the filtered isopropyl alcohol prior to filtering the sample.

## 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.

6.2 Acquisition requirements. Acquisition documents must specify the following:

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- a. Title, number, and date of this specification.
- b. Type and capacity of containers.
- c. Quantity by weight in pounds.
- d. When a different sampling plan is required (4.2).
- e. Packaging requirements (5.1).

6.3 Safety Data Sheets. Contracting officers will identify those activities that require copies of completed Safety Data Sheets (SDS) prepared in accordance with the 29CFR1910.1200.

6.4 Subject term (key word) listing.

Fuel

N-nitrosodimethylamine

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

### UDMH ASSAY, WATER AND AMINES

#### A.1 SCOPE

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

#### A.2 METHOD

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

A.2.1.1 Procedure. A 60 meter (m) x 0.53 millimeters (mm), 1.00 micrometer (µm) film thickness Agilent DB-Wax capillary column, a TCD, with a split injector and a glass wool packed split liner shall be used. Table A-1 provides a summary of the critical operating parameters. These parameters may vary depending on GC system, detector brands, and column length and may be adjusted to optimize component resolution.

**TABLE A-1. GC and method parameters**

PARAMETER	CONDITION
Injector Port Temp.	150 °C
Injection Mode	Split injection
Sample Volume	3 µL
Split Ratio	1:25
Carrier Gas	He
Control Flow	10 mL/min
Control Mode	Flow
TCD Detector Temp.	210 °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 µL of sample. The checks must meet the quality control criteria in A.2.1.2.

The elution order of possible sample components is as follows:

Air > ammonia > dimethylamine/methylamine (co-elute) > UDMH > water

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## APPENDIX A

A.2.1.2 Quality control. The following performance criteria must be met for fuel purity analysis,

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

A.2.1.3 Calculations. Calculate the percent by weight of the component detected in the sample as follows:

$$\%C = \frac{A_c}{\sum A_i} \times 100$$

where,

$A_c$	=	The measured area of the DMA/MA peak multiplied by its signal attenuation factor.
$\sum A_i$	=	The sum of all the measured peak areas multiplied by their respective signal attenuation factors
$\%C$	=	The weight percent of the component corresponding to $A_c$ .

Report amines in weight percent as,

$$Wt\% \text{ Amines} = Wt\% \text{ DMA/MA}$$

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

A.2.1.4 Equipment.

- Gas chromatograph equipped with a thermal conductivity detector (TCD) and a split injector.
- Agilent ChemStation or equivalent.
- A 60 m x 0.53 mm ID and 1 µm film thickness DB-wax capillary column (Agilent P/N 125-7062 or equivalent).
- Glass wool packed split liner (Restek P/N 23309 or equivalent).

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## APPENDIX B

### N-NITROSODIMETHYLAMINE

#### B.1 SCOPE

B.1.1 Scope. This Appendix describes a method for the determination of NDMA, content in the propellant. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

#### B.2 METHOD

##### B.2.1 Gas chromatographic method.

B.2.1.1 Procedure. A 60 m x 0.53 mm x 1 µm film thickness Stabilwax®-DB megabore column, a flame ionization detector (FID), a split injector and a deactivated glass wool split liner shall be used. GC parameters are listed in Table B-1. The analysis requires approximately 14 min for elution of all components.

**TABLE B-1. GC Conditions**

PARAMETER	CONDITION
Injector Port Temp.	150°C
Injection Mode	Split injection
Sample Volume	3 µL
Split Ratio	1:50
Carrier Gas	He
Column Flow	10 mL/min
Control Mode	Flow
FID Detector Port Temp.	210 °C
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 ( $t_{f2}$ )	2.75 min

B.2.1.2 Calibration. Prepare a calibration standard equivalent to 100 ppm NDMA by injecting 100 µL of analytical grade NDMA into a 1 L volumetric flask containing approximately 500 mL purified water. Shake the mixture, dilute to the mark with purified water, and mix the solution thoroughly. Alternatively, a certified commercially obtained solution may be used and diluted accordingly. If NDMA is found in the propellant sample, analyze the standard and sample(s) in triplicates; the RSD ≤ 15%.



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## APPENDIX B

B.2.1.3 Calculation. Use the following formula to calculate the NDMA content of the propellant:

$$NDMA\ Wt\% = \left[ \frac{Std\ conc \left( \frac{A_s}{A_{Std}} \right)}{D_{UDMH}} \right] 100$$

where

*Std conc* = NDMA Standard concentration, µg/µL

*A<sub>s</sub>* = Area of the sample NDMA peak times its signal attenuation factor.

*A<sub>Std</sub>* = Area of the standard NDMA peak times its attenuation factor.

*D<sub>UDMH</sub>* = Density of UDMH, 790.8 µg/µL

B.2.1.4 Reagents and equipment.

B.2.1.4.1 Reagents.

a. NDMA Analytical standard, certified.

B.2.1.4.2 Equipment.

- a. Gas chromatograph equipped with a flame ionization detector (FID) and a split injector.
- b. Agilent ChemStation or equivalent.
- c. A 60 m x 0.53 mm ID and 1 µm film thickness Stabilwax®-DB capillary column (Restek catalog No. 10858 or equivalent).
- d. Glass wool packed split liner (Restek P/N 23309 or equivalent).

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## CONCLUDING MATERIAL

Custodians:  
Navy – AS  
Air Force – 68  
DLA – PS

Preparing Activity  
Air Force – 68  
(Project: 9135-2013-004)

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