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October 18, 1994

PROPELLANTS, RECOVERED HYDRAZINE FAMILY FUELS, SPECIFICATION FOR

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Space Administration

John F. Kennedy Space Center

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PROPELLANTS, RECOVERED HYDRAZINE FAMILY FUELS, SPECIFICATION FOR

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JOHN F. KENNEDY SPACE CENTER

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ABBREVIATIONS AND ACRONYMS

ANSI	American National Standards Institute
API	American Petroleum Institute
ASTM	American Society for Testing
CCAS	Cape Canaveral Air Station
CFR	Code of Federal Regulations
DOT	Department of Transportation
F.S.	full scale
GC	gas chromatograph
HS	hydrazine solution
IM	Installation Management and Operations Directorate
IPA	isopropyl alcohol
JPC	Joint Propellants Contractor
KOH	potassium hydroxide
kg/L	kilograms per liter
kPa	kilopascals
KSC	Kennedy Space Center
L	liter
MIL	military
MHF	mixed hydrazine fuel
MHS	methylhydrazine solution
MPE	Materials and Processes Engineer
mg/L	milligrams per liter
mL	milliliter
mm	millimeter
MMH	monomethylhydrazine
n/a	not applicable
O.D.	outside diameter
psi	pounds per square inch
STD	standard
SPEC	specification
UDMH	unsymmetrical dimethylhydrazine
USAF	United States Air Force
µL	microliter
°C	degree Celsius
°F	degree Fahrenheit

PROPELLANTS, RECOVERED HYDRAZINE FAMILY FUELS, SPECIFICATION FOR

1. SCOPE

This specification covers the requirement for three different grades of the hydrazine family fuel blends recovered from propellant systems. This specification provides a means of categorizing recovered propellants for either reclamation for alternate reuse or for reprocessing to original manufacturing purity. This document is mandatory for use by all NASA organizations and programs of the Kennedy Space Center (KSC) and the United States Air Force (USAF) at the Cape Canaveral Air Station (CCAS).

2. APPLICABLE DOCUMENTS

The following documents form a part of this document to the extent specified herein. When this document is used for procurement, including solicitations, or is added to an existing contract, the specified revision levels, amendments, and approval dates of said documents shall be specified in an attachment to the Solicitation/Statement of Work/Contract. In the event of conflict between the documents referenced herein and the contents of this specification, the contents of this specification shall be considered a superseding requirement.

2.1 Governmental.

2.1.1 Specifications.

John F. Kennedy Space Center (KSC), NASA

KSC-C-123	Surface Cleanliness of Fluid Systems, Specification for

Federal

TT-I-735	Isopropyl Alcohol

Military

MIL-P-25604	Propellant, UNS-Dimethylhydrazine
MIL-P-26536	Propellant, Hydrazine
MIL-P-27401	Propellant Pressurizing Agent, Nitrogen

MIP-P-27404	Propellant, Monomethylhydrazine
MIL-P-27407	Propellant Pressurizing Agent, Helium

2.1.2 Standards.

Military

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

2.1.3 Code of Federal Regulations (CFR).

49 CFR 170-190	Department of Transportation Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles

[Copies of specifications, standards, drawings, and publications required by suppliers in connection with specified procurement functions should be obtained from the procuring activity or as directed by the Materials and Processes Engineer (MPE).]

2.2 Non-Governmental.

American National Standards Institute (ANSI)

ANSI Z129.1-88	Hazardous Industrial Chemicals, Precautionary Labeling

(Application for copies should be addressed to the American National Standards Institute, 1420 Broadway, New York, NY 10010.)

American Society for Testing and Materials (ASTM)

ASTM E29	Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
ASTM D2276-93	Standard Test Method for Particulate Contaminant in Aviation Fuel Line Sampling

ASTM D287 — Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

(Applications for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Chemical and Physical Properties. - The chemical and physical properties of the recovered propellants shall conform to the grades listed below and in table 1 when tested in accordance with the applicable test methods (4.5).

Grades include:

a. Methylhydrazine Solution (MHS)

b. Hydrazine Solution (HS)

c. Mixed Hydrazine Fuel (MHF)

Table 1. Chemical Properties

Composition	MHS	HS	MHF	Test Paragraph
Monomethylhydrazine (% by wt)	65 min	N/A	Report	4.5.4
Hydrazine (% by wt)	N/A	65 min	Report	4.5.4
UDMH (% by wt)	N/A	N/A	Report	4.5.4
Total N_2H_4, MMH, UDMH, & Alcohol	N/A	N/A	75 min	4.5.5
Alcohol (% by wt)	0.02 max	0.02 max	10 max	4.5.6
Water (% by wt)	35 max	35 max	25 max	4.5.4
Particulate (mg/L)	10 max	10 max	20 max	4.5.2
Density (kg/L)	Report	Report	Report	4.5.3

3.2 Limiting Values. - The following statement 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 righthand place

of figures used in expressing the limitation value, in accordance with the rounding off method of ASTM E29.

3.3 Filter. - A filter with a 10-micron nominal and a 40-micron absolute rating shall be used whenever MHS, HS, or MHF is transferred into portable tanks or cargo tanks.

3.4 Qualitative Requirements. - HS and MHS propellants shall be clear, water-white homogeneous liquids when examined visually by transmitted light. A slight color tint, especially yellow to amber, may be observed in mixed hydrazine fuel propellants. Any tint and the presence of particulates shall be noted on the analysis sheet.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. - Unless otherwise specified in the contract, the KSC/CCAS Joint Propellants Contractor (JPC) is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the JPC may use its 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 ensure that 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 Quality Conformance Tests. - Quality conformance tests shall consist of:

a. Individual Tests (4.3.1)

b. Sampling Tests (4.3.2)

4.3.1 Individual Tests. - The propellant shall be sampled by the Examination of Product test described in 4.5.1.

4.3.2 Sampling Tests. - The propellant shall be selected in accordance with 4.3.2.1 and subjected to the following tests:

a. Particulate (4.5.2)

b. Density (4.5.3)

c. Amines assay, water, and isopropyl alcohol (IPA) (4.5.4)

d. Total amines and isopropyl alcohol (4.5.5)

e. Alcohol in HS and MHS (4.5.6)

4.3.2.1 Sampling Plan.

4.3.2.1.1 Lot. - A lot shall consist of the following:

a. The propellant recovered from individual runs of a batch process which is used to fill recovery containers directly from the process output.

b. The recovered propellant is consolidated into a larger volume from recovery containers filled in a batch process (a. above) to form a new homogeneous mixture.

4.3.2.1.2 Sample. - A sample consists of not less than 600 milliliters (ml) of propellant. Unless otherwise specified, a quality conformance test shall be made on each required sample of the propellant as it is taken directly from the recovery or consolidation containers. The bottles intended for sampling shall be specially cleaned and handled in accordance with the procedure described in KSC-C-123, Level 50A.

4.3.2.1.3 Drums. - Drum sampling shall be at the discretion of JPC Fluids Management if the individual recovery drums are each provided with an identification tag per 5.4.1 of the propellant contained therein. Drums whose contents, concentrations, and/or origins are not otherwise known, shall each be considered a lot and shall be individually sampled.

4.3.2.1.4 Portable Tanks and Cargo Tanks. - Each portable tank or cargo tank shall constitute a lot. JPC Fluids Management may defer tank sampling until an adequate inventory of recovered propellant has been accumulated in that tank. This will preclude repetitive sampling of partial tank loads of recovered propellants.

4.3.2.1.5 Other Containers. - Unless otherwise specified, other containers of 378 liters (100 gallons) or less water capacity shall be sampled in accordance with 4.3.2.1.3 and other containers greater than 378 liters water capacity shall be sampled in accordance with 4.3.2.1.4.

4.4 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 the particulate test (4.5.2) to determine compliance with the requirement specified herein. An

examination to ensure the material conforms with the requirements specified in 3.4 shall be conducted after the sample has been transferred to the 500 mL graduated cylinder.

4.5.2 Particulate. - The propellant sample shall be tested for contamination per ASTM D2276, Method A, with the following exceptions. Report results in mg/L units.

a. Mix the sample thoroughly by shaking the sample into a clean 500 mL graduated cylinder. Use this 500 mL of propellant for the particulate analysis.

b. Use a solvent resistant filter disc made from such materials as Millipore LCWP 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 ASTM D2276.

c. Filtered isopropyl alcohol shall be used for rinsing the sample bottle and filter holder instead of petroleum ether specified in ASTM D2276. Water should not be used.

d. Save the filtered propellant for determining density (see 4.5.3).

4.5.3 Density. - The propellant density shall be determined by the general procedure described in ASTM D287. Density shall be measured at 25 degrees Celsius (°C) [77 degrees Fahrenheit (°F)] and reported in kg/L units. The analyst shall make whatever changes necessary to the procedure commensurate with the hazards of hydrazine family fuels. Use up to approximately 250 mL of the propellant left over from the particulate analysis in 4.5.2.

4.5.4 Amines Assay, Water, and Isopropyl Alcohol. - The propellant assay, isopropyl alcohol, and water content shall be determined by the following method.

4.5.4.1 Column Conditioning. - Use a column consisting of 4 percent Carbowax 20 M 60/80 Carbopack B with 0.8 percent KOH, packed into a 0.32 mm (0.125 inch) O.D. by 1.3 m (6 feet) long stainless steel tube. This column packing is commercially available from Supelco in accordance with Gas Chromatograph (GC) Bulletin 737E, or an equivalent packing material may be used. Condition the column with carrier gas flowing and the oven set at 120 °C (248 °F) for one hour. After conditioning the column, connect the other end to the detector and set the carrier gas flow to approximately 25 mL/min, and the column over to 100 °C (212 °F). The inlet and detector temperatures, if separately heated, shall be set to 100 °C and 150 °C (302 °F), 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.

4.5.4.2 Analysis. - Equilibrate the column with propellant by injection of two or more 0.5 microliter (μL) samples into the inlet. If more than 30 minutes elapse between analyses, a single 5 μL injection of propellant should reequilibrate the column. Inject 0.1-0.2 μL of propellant for analysis and record the areas of all peaks in the chromatogram. Each analysis should require less than 10 minutes for elution of all components. The elution order of possible sample components is as follows: water, hydrazine, MMH, UDMH and IPA.

4.5.4.3 Calculations. - The following formula shall be used to calculate the percent by weight of each component appearing in the chromatogram.

$$\%C = \frac{Ac}{\Sigma Ai} \times 100$$

where Ac = the measured area of a peak multiplied by its signal attenuation factor.

ΣAi = the sum of all of the measured areas multiplied by their respective signal attenuation factors.

%C = the weight percent of the component corresponding to Ac.

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

4.5.4.4 Equipment and Reagents. - The following equipment and reagents shall apply as test conditions of 4.5.4.

a. Equipment

1. Gas Chromatograph (GC): equipped with a thermal conductivity detector.

2. Integrator, electronic.

3. Hypodermic syringe: 10 microliter, fixed needle.

4. Regulator: helium, to fit the cylinder.

b. Reagents

1. GC 737E, Supelco or equivalent.

2. Helium gas: conforming to MIL-P-27407.

4.5.4.5 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), UDMH (MIL-P-25604), and MMH (MIL-P-27404). The composition of the mixture should be approximately 40 percent MMH, 30 percent N_2H_4, 10 percent UDMH, 10 percent IPA, and 10 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, MMH, UDMH, and finally IPA. Calculate the actual composition as described in 4.5.4.3.

4.5.5 Total Amines and Alcohol. - The weight percent of N_2H_4, MMH, UDMH and alcohol determined in 4.5.4.3 shall be individually reported and then added together and reported as percent MHF.

4.5.6 Alcohol. - Alcohol shall be determined in MHS and HS by the following method.

4.5.6.1 Procedure. - Use the column consisting of 10 percent UCON 50 HB-5100 and 3 percent KOH coated upon Chromosorb WAW (80/100) mesh, packed into a 0.32 mm (0.125 inch) O.D. by 1.3 m (6 feet) long stainless steel tube. Use a column oven temperature of 80 °C (176 °F). This column packing is available commercially from Analabs Inc. as packing No. 260, or equivalent packing material may be used. A flame ionization detector shall be used. The order of separation is ammonia, UDMH, isopropanol, MMH, followed by hydrazine. Ammonia which emerges prior to the UDMH peak shall not be summed with the carbonaceous material. Allow enough time between injections for the baseline to stabilize before injecting a second series of samples.

4.5.6.2 Calibration. - Add 9.4 µL of IPA, 9.4 µL of UDMH, and 8.3 µL of MMH to 75 mL of propellant sample contained in a 118 mL (4 oz) screw-capped bottle, cap, and mix the solution thoroughly. Transfer a portion of this calibration mixture to a sample vial. Analyze both samples and record the areas of the ammonia, isopropyl alcohol, UDMH, and MMH peaks.

4.5.6.3 Calculations. - The combined UDMH/alcohols peak shall be calculated as percent IPA. MMH is calculated separately.

$$\% \text{ IPA} = \frac{0.01\ Ai}{Aci - Ai}$$

Where: Ai = Area of sample IPA times its attenuation factor

Aci = Area of calibration IPA times its attenuation factor

$$\%\text{MMH} = \frac{0.01\ Am}{Acm - Am}$$

Where: Am = Area of sample MMH times its attenuation factor

Acm = Area of calibration MMH times its attenuation factor

$$\%UDMH = \frac{0.01\ Au}{Acu - Au}$$

Where: Au = Area of sample UDMH times its attenuation factor

Acu = Area of calibration UDMH times its attenuation factor

Reporting:

% Alcohol = %IPA + %MMH + %UDMH for Hydrazine Solution (HS)

% Alcohol = %IPA for Methylhydrazine Solution (MHS)

NOTE: Peak height may be substituted for peak area providing peak is sharp.

4.5.6.4 Reagents and Equipment. - The following reagents and equipment shall apply as test conditions of 4.5.4.

4.5.6.4.1 Reagents.

a. UDMH = conforming to MIL-P-25604

b. MMH = conforming to MIL-P-27404

c. IPA = conforming to TT-I-735A

d. Reagents = as required in 4.5.5

e. Reagents: 10 percent UCON 50 HB-5100 and 3 percent KOH coated upon Chromosorb WAW (80/100) mesh. Analabs No. 260 or equivalent.

4.5.6.4.2 Equipment. - The following equipment shall apply as test conditions of 4.5.4.

a. Gas chromatograph - incorporating a flame ionization detector.

b. Recorder - potentiometric strip chart, 0-1 millivolt, 1 second full scale (F.S.) response, with integrator.

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.

5. PREPARATION FOR DELIVERY

5.1 Packaging. - The propellant shall be packaged in containers as specified by the JPC. Packaging shall comply with the requirements of the Department of Transportation (DOT) Regulations 49 CFR 170-190, or a waiver from the KSC/CCAS Safety Organizations, obtained by the JPC.

5.2 Preparation of Containers. - The JPC shall establish the condition of all containers to ensure that they are free from contamination and suitable for shipment and storage. Leased or government owned containers shall be cleaned and repaired in accordance with the schedule established in the contract. Internal inspections on cargo tanks or portable tanks used in exclusive continuous service need be made only upon initial entry into that service or at any required retest or overhaul.

5.2.1 Cleaning and Repair. - Unless otherwise provided for in the JPC contract, any physical damage to containers which would endanger safe transportation of the propellant shall be repaired prior to reuse. If evidence of excessive internal particulate contamination is found, the containers shall be recleaned by a suitable method to remove the contamination.

5.2.2 Gaskets. - Gaskets used to seal container openings shall be polyterafluorethylene or other material compatible with the propellant. The contractor shall ensure that all gaskets are serviceable and furnish new gaskets when necessary so that a tight seal is ensured.

5.3 Filling. - Containers shall not be entirely filled. A minimum 10 percent-by-volume ullage space shall be left in each container to ensure that no leakage or over-pressurization of the container occurs. After filling of containers, the space above the liquid level shall be filled with nitrogen conforming to MIL-P-27401, Type I, to not less than 34.4 kPa (5 psi) or as specified in the container's operating procedure.

5.4 Labeling and Marking. - Each container shall be marked, labeled and placarded in accordance with DOT regulations. 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, an identification tag, KSC Form 28-689, shall be secured to each container and shall contain the following information: Propellant name, quantity, facility of origin, name of generating contractor or program, and date of recovery from a propellant system.

5.4.2 Precautionary Label. - A precautionary label prepared in accordance with ANSI Standard Z 129.1 shall be applied to each container.

5.4.3 Container Color Code. - Unless otherwise specified by the procuring activities, each container shall be color coded in accordance with MIL-STD-172.

6. NOTES

6.1 Intended Use. - The recovered propellants which conform to this specification are intended for use as follows:

MHS	Reprocess to conform to MIL-P-27404B.
HS	Reprocess to conform to MIL-P-26536D.
MHF	Use as a fuel for conducting hypergolic or air-fed hydrazine family fuel fire training exercises.

6.2 Definitions. - For the purpose of this specification, the following definitions shall apply.

a. Particulate. The undissolved solids retained on a 10 micron filter membrane.

b. 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. KSC/CCAS propellants users are enjoined to approach the appropriate pollution control district to mutually resolve all problems areas, and to develop adequate control and disposal methods for situations which are likely to develop in any of the phases.

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