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MILITARY SPECIFICATION PROPELLANT, HYDRAZINE

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 three grades of hydrazine (N2 H4).
- 1.2 Classification. The hydrazine covered by this specification shall be of the following grades, as specified (6.4).

GRADES

Standard

Normal production and quality control.

suitable for most uses.

Monopropellant

Normal product with strict control of specified impurities. To be specified only for monopropellant catalytic engines where extended life is a

requirement.

High Purity

Special production with strict control

of specified impurities.

2. APPLICABLE DOCUMENTS

- 2.1 Government Documents.
- 2.1.1 Specifications and Standards. The following specifications and standards form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, noted in the solicitation.

Beneficial Comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Code (68) SA-ALC/SFRM, Kelly AFB TX 78241-5000, by using the self-addressed standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter. AMSC N/A FSC 9135

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SPECIFICATIONS

MILITARY

MIL-P-27401

Propellant, Pressurizing Agent, Nitrogen

MIL-P-27407

Propellant, Pressurizing Agent, Helium

STANDARDS

MILITARY

MIL-STD-129

Marking for Shipment and Storage

MIL-STD-172

Color Codes for Containers of Liquid

Propellants

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

2.1.2 Other Government Documents. The following other Government documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issues shall be those in effect on the date of the solicitation.

Department of Transportation (DOT)

49CFR 100-199 TRANSPORTATION

P 5800.3 EMERGENCY RESPONSE GUIDEBOOK

(The Code of Federal Regulations (CFR) and the Federal Register are for sale on a subscription basis by the superintendent of Documents, US Government Printing Office, Washington DC 20402. When indicated, reprints of certain regulations may be obtained from the federal agency responsible for issuance thereof.)

2.2 Other <u>Publications</u>. The following non-government documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted shall be those listed in the issue of the DODISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS shall be the issue of the non-government documents which is current on the date of the solicitation.

American National Standards Institute (ANSI)

ANSI-Z129.1

Chemicals, Hazardous Industrial, Precautionary Labeling of

(Application for copies of ANSI publications should be addressed to the American National Standards Institute, 1420 Broadway, New York, NY 10018.)

American Society for Testing and Materials (ASTM)

ASTM D 2276 Fuels, Aviation Turbine, Particulate Contaminant in

ASTM E 29 Indicating which of places of figures are to be considered significant in specified limiting values

ASTM D 1193 Specification for Reagent Water

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

American Chemical Society (ACS) Specification

ACS Reagent Chemical Standards

(Application for copies should be addressed to the American Chemical Society, 1155 16th St NW, Washington DC 20036.)

- 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.
- 2.3 Order of Precedence. In the event of a conflict with the text of this specification and the references cited herein, (except for associated detail specifications, specification sheets or MS standards) the text of this specification shall take precedence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

REQUIREMENTS

3.1 <u>Chemical and Physical Properties</u>. 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 I. CHEMICAL AND PHYSICAL PROPERTIES

PROPERTIES	LIMITS			
	STANDARD GRADE	MONOPROPELLANT GRADE	HIGH PURITY GRADE	TEST PARAGRAPH
Hydrazine (percent by wt)	98 min	98.5 min	99.0	4.5.2
Water (percent by wt)	1.5 max	1.0 max	1.0 max	4.5.2
Ammonia (percent by wt)			0.3	4.5.2
Particulate (milligrams per liter)	10 max	1.0 max	1.0 max	4.5.3
Chloride (percent by wt)		0.0005 max	0.0005 max	4.5.4
Aniline (percent by wt)		0.50 max	0.003 max	4.5.5
Iron (percent by wt)		0.002 max	0.0004 max	4.5.6
Nonvolatile Residue (percent by wt)		0.005 max	0.001 max	4.5.7
Carbon Dioxide (percent by wt)		0.003 max	0.003 max	4.5.8
Other Volatile Cabonaceous Material (Total as MMH & UDMH, Alcohol, percent by wt)		0.02 max	0.005 max	4.5.9

3.2 <u>Limiting Values</u>. The following applies to all specified limits in this specification. For purposes of determing 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).

3.3 <u>Filter</u>.

3.3.1 Standard Grade. A filter with a 10-micrometer nominal and 40-micrometer absolute rating shall be installed between the manufacturer's plant system and the container to be filled for delivery.

- 3.3.2 Monopropellant and High Purity Grades. A filter with a 2-micrometer nominal and 10-micrometer 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 colorless, 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 <u>Classification of Tests</u>. 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 tests 4.4.1
 - b. Sampling tests 4.4.2
- 4.4.1 <u>Individual Test</u>. The propellant shall be subject to the following test as described under 4.5.
 - a. Examination of product 4.5.1
- 4.4.2 <u>Sampling Tests</u>. The propellant shall be selected in accordance with 4.4.2.1 and subjected to the tests indicated in Table I as described under 4.5.
 - 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 issued 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 without 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 <u>Sample</u>. A sample consists of not less than 600 ml of propellant when tested in accordance with standard grade requirements and not less than 1500 ml when tested in accordance with monopropellant grade and high purity grade requirements. Unless otherwise specified, quality conformance tests shall be made on the sample of 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 bottle intended for sampling shall be specially cleaned and handled in accordance with procedure described in 4.5.3. The sampling of drums and other shipping containers shall be conducted in such a manner that neither the contents of the container nor the sample is exposed to the atmosphere. The use of a plastic bag purged with nitrogen, to cover the container, is mandatory.
- 4.4.2.1.3 <u>Drums</u>. 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.

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

Table II. SAMPLING FOR TEST

4.4.2.1.4 <u>Portable Tanks, Cargo Tanks, and Tank Cars</u>. 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.
- 4.4.4 <u>Chemicals</u>. Unless otherwise specified, all chemical tests shall be made with ACS specification reagent grade chemicals and ASTM D-1193, Type III, Reagent Water.

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 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 calibrated cylinder for standard grade or to a 1000 ml graduated cylinder for monopropellant and high purity grades.
- 4.5.2 Hydrazine 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 Assay Column Preparation. Cap one end of a 1/8 inch 0.D. by 6-foot nickel tube and fill the tube with the Tenax GC 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 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 ml/min, and the column oven to 50°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.
- 4.5.2.1.2 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 analysis, 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, using the following time/temperature program:

He carrier gas flow 20-25 ml/min. Initial temperature 50°C Rate 2°C/min. Final Temperature 155°C Final time hold 4 min.

Each analysis should require less than 20 minutes for elution of all components. The elution order of possible sample components is as follows:

air, ammonia, methylamine, water, monomethylhydrazine, and hydrazine

4.5.2.1.3 Calculations.

$$\chi_{N_2H_4} = \frac{A_{N_2H_4} \times 100}{\Sigma A_1}$$

$$\chi_{H_2O} = \frac{A_{H_2O} \times 100}{\Sigma A_1}$$

$$\chi_{NH_3} = \frac{A_{NH_3} \times 100}{\Sigma A_1}$$
 for high purity grade only

WHERE:

A _{N2} H ₄	s	The measured area of the N_2H_4 peak multiplied by its signal attenuation factor.
A _{H2} 0	z	The measured area of the $\rm H_2O$ peak multiplied by its signal attenuation factor.
A _{NH3}	£	The measured area of the NH_3 peak multiplied by its signal attenuation factor.
2A ₁	=	The sum of all the measured peak areas times their signal attenuation factors.

NOTE: For monopropellant and high purity grades substitute $(\Sigma A_1)(100/[100 - %aniline])$ for ΣA_1 to correct for the aniline content of the propellant.

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.

b. Reagents:

Tenax G. C. 60/80 mesh, Analabs, Inc., 80 Republic North Haven CT 06473 or equivalent.

- 4.5.3 <u>Particulate</u>. The propellant sample shall be tested for contamination in accordance with ASTM Designation D-2276, Method A, with the following exceptions.
- 4.5.3.1 Mix the sample thoroughly without exposure to air. Immediately pour 500 ml of the sample into a clean 500 ml graduated cylinder. Use this 500 ml of propellant for the particulate analysis of standard grade. For monopropellant and high purity grades substitute a 1000 ml graduated cylinder and use 1000 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) or Gelman VF-6, (Fluoride-Metricel), plain, white, 10+3 microns, 47 mm diameter, or equivalent, for standard grade and a Millipore FALP04700, plain, white, 1 micron, 47 mm diameter or equivalent for monopropellant and high purity grades instead of that specified in ASTM D-2276.
- 4.5.3.3 The drying oven temperature shall be 158°F (70°C) instead of the 194°F (90°C) specified in ASTM D-2276.
- 4.5.3.4 Filtered isopropyl alcohol shall be used for rinsing the sample bottle and filter holder instead of petroleum ether specified in ASTM D-2276.
- 4.5.4 <u>Chloride</u>. The Chloride level shall be determined by methods based on accepted scientific (chemical) principles, such as colorimetric with mercury thiocyanate, combinations of silver nitrate and/or specific ion electrodes (Ag⁺/Cl⁻), or ion chromatographic techniques. In case of dispute 4.5.4.1 shall be the referee method.
- 4.5.4.1 Procedure. Rinse the propellant residue container in one vessel from 4.5.7 with a 5 ml portion of 1 N-H $_2$ SO $_4$. Briefly stir and heat the liquid to boiling but do not allow the condensate to escape. Transfer the solution to a 25 ml volumetric flask. Rinse vessel with a second 5 ml portion of acid followed by one 5 ml portion of distilled water. Combine the portions in the 25 ml volumetric flask and add two to three drops of Bromcresol Green indicator. Dropwise titrate the contents of the flask to a green endpoint with 1 N-NaOH and then dilute to the mark with distilled water. Mix the solution and transfer approximately 10 ml to a 25 ml beaker. Immerse the electrodes in the

sample solution and record the millivolt reading. Compare the millivolt reading from the sample to a calibration curve prepared from chloride standards and record the chloride concentration of the sample.

4.5.4.3 <u>Calculation</u>. Calculate the percent chloride in the sample using the following formula.

$$%C1^{-} = \frac{ppm C1^{-} (from curve) \times 25}{1.01 \times 10^{6}}$$

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

4.5.4.4.1 Reagents.

- a. Chloride-free distilled water; the water is considered chloride-free if a 100 ml sample used to dissolve 0.1g of silver nitrate, shows no turbidity after standing in the dark for 24 hours.
- b. $0.1~N-H_2~SO_4$; prepare by pouring 3 ml of ACS grade concentrated H_2SO_4 into 300 ml of chloride-free water contained in a 1000 ml volumetric flask. Cool the solution to room temperature, dilute to the mark with the chloride-free distilled water, and mix thoroughly. Transfer the solution to a plastic bottle.
- c. 1 N-NaOH; prepare by dissolving 40 g of ACS grade, low carbonate, NaOH in 300 ml of chloride-free water contained in a 1000 ml volumetric flask. Cool the solution to room temperature, dilute to the mark with chloride-free distilled water, and mix thoroughly. Transfer the solution to a plastic bottle and protect from prolonged exposure to the atmosphere.

4.5.4.4.2 Equipment.

- a. Chloride Electrode solid state Orion Model 94-17, or equivalent.
- b. Reference Electrode, Double Junction, Orion Mode 90-02 with 1 M-KNO₃ outer compartment filling solution, or equivalent.
 - c. pH/Millivolt meter Orion Ion Analyzer Model 801, or equivalent.
- 4.5.5 Aniline. The aniline content of the propellant shall be determined by one of the following methods. 4.5.5.1 is the referee method for monopropellant grade hydrazine. 4.5.5.3 is the referee method for high purity grade hydrazine.

4.5.5.1 Spectrophotometric Method.

4.5.5.1.1 Procedure. Pipet or syringe 0.25 ml (250 microliters) of propellant into a 25 ml volumetric flask containing approximately 15 ml of

distilled water, dilute to the mark with distilled water, and mix the solution thoroughly. Fill two 1 cm pathlength silica spectrophotometer cells with distilled water and place one in each position for sample and reference cells. Using the appropriate source and detector for the spectrophotometer set the recorder pen to zero absorbance at 320 nm and scan the region between 320 nm and 240 nm. If the baseline is relatively flat then the cells and distilled water can be considered free from interfering substances. Exchange the water in the sample cell with the previously prepared sample, using several rinses with sample solution, and rescan the 320 nm to 240 nm region, first insuring that the recorder pen is set at zero absorbance at 320 nm. Draw a baseline under the aniline peak at 280 nm, measure, and record the net absorbance of the peak. Determine the aniline content of the sample by comparing the net absorbance obtained to a calibration curve of net absorbance versus percent aniline.

- 4.5.5.1.2 Calibration. Pipet or syringe 0.025 ml (25 microliters) of aniline into a 100 ml volumetric flask containing 50 ml of distilled water. Shake the mixture to dissolve the aniline and dilute the solution to the mark with distilled water. Mix the standard thoroughly. Transfer to separate 25 ml volumetric flasks, the following volumes of the previously prepared aniline standard; 2.0 ml, 4.0 ml, 6.0 ml, and 8.0 ml. Dilute each to the mark with distilled water and mix thoroughly. The aniline concentrations are equivalent to 0.2, 0.4, 0.6, and 0.8% aniline in propellant. Determine the net absorbance of standards versus distilled water in the same manner as with the sample. Plot net absorbance versus percent aniline and draw a curve through the points. All calibrations standards should be prepared fresh daily.
- 4.5.5.1.3 Equipment. The following shall apply as test conditions of 4.5.5.1.
- 4.5.5.1.3.1 Equipment. Spectrophotometer, double beam, ultra violet, narrow band width, recording, equipped with 1 cm silica cells, Cary 14 or equivalent.

4.5.5.2 Gas Chromatographic Method.

- 4.5.5.2.1 Procedure. Prepare a gas chromatographic column consisting of 9.5 g Apiezon L and 0.5 g Amine 220 coated upon 20 g of a white, silanized, diatomaceous earth support 60/80 mesh. Condition the column for four hours at 150°C prior to connection to the detector. Use a gas chromatograph equipped with a flame ionization detector. The inlet, if separately heated, should be adjusted to 130°C or greater, the column and detector temperatures should be 130°C and 150°C, respectively. After the instrument has stabilized, inject 0.5 microliters of propellant and record the area of the aniline peak. Compare the area of the aniline peak with that of a calibration standard and calculate the percent aniline of the sample.
- 4.5.5.2.2 <u>Calibration (monopropellant hydrazine)</u>. Pipet or syringe 0.5 ml (500 microliters) of aniline into a 100 ml volumetric flask containing

approximately 50 ml of distilled water, shake the mixture, dilute to the mark with distilled water, and mix the solution thoroughly. This calibration standard is equivalent to 0.5% aniline in propellant. Inject 0.5 microliters of the standard into the inlet of the chromatograph and record the area of the aniline peak. Prepare the standard fresh daily.

4.5.5.2.3 <u>Calculation</u>. Use the following formula for calculation of the aniline content of the propellant.

% aniline =
$$\frac{0.5A_S}{A_C}$$

WHERE:

As = Area of the sample aniline peak times its attenuation factor.

A_C = Area of the standard aniline peak times its attenuation factor.

- 4.5.5.2.4 Equipment. The following equipment shall apply as test conditions of 4.5.5.2.
 - a. Gas chromatograph, equipped with a flame ionization detector.
- b. Recorder, potentiometric, with integrator, pen speed = 1 sec.
 F.S., 1 mv span.
- 4.5.5.3 Wide Bore Capillary Method. A 0.53 mm 0.D. x 15 meter crosslinked "Carbowax 20M" capillary column shall be used. Helium is used as the carrier gas. A gradient temperature program is used from 40°C to 130°C. Splitless injection is used. Sample size should be 0.4 to 1 microliter. Condition the column with several injections of the calibration mixture 3 min. apart. After the fifth injection, allow sufficient time for the final injection of aniline to elude, approximately 10 min. Adjust flow rate to allow for baseline separation of the trace monomethyl hydrazine from hydrazine.
- 4.5.5.3.1 Calibration of Calculation (High Purity Hydrazine). Pipet or syringe 0.5 ml of aniline into a 1.0 l volumetric flask containing approximately 500 ml of distilled water, and mix the solution thoroughly. Pipet 10 ml of this solution into a 100 ml volumetric flask. This calibration standard is equivalent to 0.005% aniline in propellant. Inject 1.0 microliter of the standard into the inlet of the chromatograph and record the area of the aniline peak. Use a similar calculation method described in 4.5.5.2.3 allowing for the less concentrated standard solution of 0.005%.
- 4.5.6 Iron. The iron content of the propellant shall be determined by one of the following methods. 4.5.6.1 is the referee method.

4.5.6.1 Atomic Absorption.

- 4.5.6.1.1 Procedure. Rinse the propellant residue contained in one vessel from 4.5.7 as follows. Add 5 ml of 5 N-H $_2$ SO $_4$ to the vessel and place on a hotplate stirrer. Heat the mixture, while stirring, to boiling and maintain at that temperature for three to five minutes. Allow the solution to cool and transfer to a 100 ml volumetric flask. Rinse the vessel at least three times with 5 ml portions of 0.1 N-H $_2$ SO $_4$ combining all of the rinses in the volumetric flask. Dilute to the mark with distilled water and mix thoroughly. (For atomic absorption only, hydrazine may be diluted 2.1 and used directly.) Determine the absorbance of the sample at an iron wavelength closest to 2480A on a suitable atomic absorption spectrophotometer, after setting the instrument to zero absorbance, with 0.1 N-H $_2$ SO $_4$. Compare the results to a calibration curve prepared from iron standards and record the iron-concentration of the sample.
- 4.5.6.1.2 <u>Calibration</u>. Prepare a 1000 ppm iron standard by dissolving 1.00 g of iron wire or shot in 50 ml of 6 N-H₂SO₄. Use heat. Quantitatively transfer the resulting solution to a 1000 ml volumetric flask with 0.1 N-H₂SO₄ and dilute to the mark with 0.1 N-H₂SO₄. To four 200 ml volumetric flasks add successively 1.0, 2.0, 3.0, 5.0 ml of the iron standard, dilute each to the mark with 0.1 N-H₂SO₄, and mix thoroughly. The iron concentrations of these solutions are 5, 10, 15, and 25 ppm, respectively. (Commercial standard solutions may be used.) After setting the instrument to zero absorbance with 0.1 N-H₂SO₄, determine the absorbance of the four calibration standards. Prepare a calibration curve of absorbance versus ppm iron.
- 4.5.6.1.3 <u>Calculation</u>. Calculate the iron content of the propellant using the following formula.

%Fe =
$$\frac{(ppm \ Fe) \times 2}{1.01 \times 10^4}$$

WHERE:

ppm Fe =

The iron content of the sample solution, using the calibration curve.

4.5.6.1.4 Equipment. the following equipment shall apply as test conditions of 4.5.6.1.

4.5.6.1.4.1 <u>Equipment</u>.

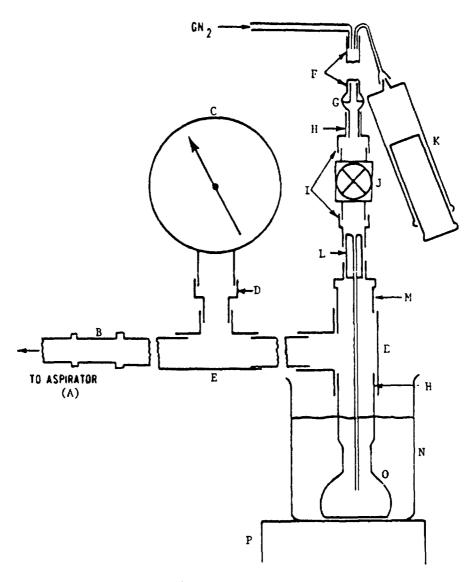
- a. Hotplate/stirrer controllable heat and stir settings.
- b. Atomic Absorption Spectrophotometer, Perkin Elmer 503 or equivalent with an iron hollow cathode lamp.

4.5.6.2 Colorimetric Method.

- 4.5.6.2.1 <u>Procedure</u>. Prepare the sample solution in the same manner as described in 4.5.6.1. Analyze the solution using the method described by ASTM D-1068, Method A, paragraph 12.
- 4.5.7 <u>Non-Volatile Residue</u>. The non-volatile residue of the propellant shall be determined in duplicate by either of the following methods (4.5.7.1 shall be the referee method).

4.5.7.1 Dropwise Method.

- 4.5.7.1.1 Procedure. Clean the unassembled apparatus with detergent and rinse thoroughly with distilled water. Dry the parts in an oven at 110°C. Assemble the apparatus as shown in Figure 1. Fill the water bath with distilled 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 and turn on the water aspirator. With the sample delivery valve closed, the pressure in the apparatus should be below 30 mm Hq. Open the sample delivery valve slowly to adjust the apparatus pressure to approximately 60 mm Hg. 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 carefully and place it near the analytical balance. After at least twenty minutes, accurately weigh the residue vessel. Record the weight. Reassemble the apparatus and close the sample delivery valve. Replace the water bath, adjust the water level to the mark, heat the water to boiling. Transfer 100 ml of propellant to the apparatus using a 100 ml volumetric pipet. With the nitrogen purge in place, turn on the water aspirator and reduce the apparatus pressure to less than 30 mm Hq. 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 mm Hg (ca. 2 drops per second). The quantity of propellant in the residue vessel should not exceed 2 to 4 ml at the equilibrium evaporation rate. Adjust the sample delivery valve to maintain the evaporation pressure at less than 60 mm Hg. After 100 ml of propellant has been evaporated (ca. 30 minutes), allow the residue vessel to purge for an additional five minutes to insure 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. Record the weight. Clean the residue vessel by refluxing 5 ml of 5 N-H₂SO₄ in the vessel for several minutes followed by several rinses with distilled 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.
- 4.5.7.1.2 <u>Calculations</u>. Calculate the nonvolatile residue of the propellant as follows:



- A WATER ASPIRATOR (glass or polyethylene)
- B CONNECTOR (polyethylene, separable, Sargent Cat. No. S-73788, or equivalent)
- C GUAGE (absolute pressure, 0-760mm range, stainless steel, Matherson Gas Co. Model No. 63-5601, or equivalent)
- D ADAPTER, SS-811-A4F
- E UNION TEE, SS-810-3 (2 each)
- F PIPET (volumetric, borosilicate glass, 100 ml capacity)
- G JOINTS (borosilicate glass, spherical, 12/5, ball, socket, and clamp)
- H FERRULES, T-814-1, T-813-1, T-4-4-1, T-403-1
- I CONNECTOR, SS-400-7-2 (2 each)

- J VALVE (stainless steel, polytetrafluoroethylene packing, Whitey Cat. No. 55, or equivalent)
- K SYRINGE (glass, 100ml capacity, with 8-12 in. polytetrafluoroethylene needle)
- L ROD (polytetrafluoroethylene or polyethylene, ½ in OD drilled and a 1/16 OD x .025 in wall tube inserted)
- M REDUCER SS-400-R-8
- N BEAKER (glass, 1000 ml capacity)
- 0 FLASK (volumetric, borosilicate glass, 25 ml capacity)
- P HOTPLATE (adjustable heat range)

NOTE: All FITTINGS, stainless steel, swagelok or equivalent (D,E,H,I,M)

$$$ \times NVR = \frac{W_1 - W_0}{1.01}$$

WHERE:

Report the averaged result of duplicate NVR determinations.

4.5.7.1.3 Equipment. The following equipment shall apply as test conditions of 4.5.7.

4.5.7.1.3.1 Equipment. (See Figure 1)

- a. Tubing Polyethylene, 1/2 inch 0.D., borosilicate glass, 1/2 inch 0.D.
 - b. Clamps hose, to fit 1/2 inch O.D. tube.

4.5.7.2 Rotary Evaporation.

4.5.7.2.1 Procedure. Dry a 250 ml round bottom flask in an oven at 110°C for one (1) hour. Allow flask to cool in a desiccator and weigh on the analytical balance. Repeat above procedure until weight remains constant to ± 2 mg. Transfer 100 ml of hydrazine into the tared 250 ml flask. Place the flask on the evaporator using a standard taper Teflon sleeve as a seal. Attach a water aspirator pump to the evaporator via a one-liter suction flask and evaporate the hydrazine to dryness. A warm bath may be placed around the rotating flask to aid evaporation.

WARNING

The contact of hydrazine vapor with metal surfaces, as in a metal rotary evaporator, must be avoided. Do not exceed 40°C during the initial evaporation phase when hydrazine liquid is present. Chemical splash proof goggles must be worn throughout the entire evaporation process.

When no more hydrazine distills over, the water bath is lowered. Final high boiling components (mainly aniline) are distilled under vaccum at temperatures up to 100°C using a hot air gun for heating. The flask is then removed and dried in an oven at 110°C for one hour and weighed as before to a constant weight.

4.5.7.2.2 Calculations.

% Non-Volatile Residue = $\frac{\text{Weight of Residue, g x 100}}{\text{V x D}}$ V = Volume of Sample, ml D = Density of Hydrazine at Measured Temperature

4.5.7.2.3 Equipment.

- a. All glass rotary evaporator, Rincho-Buchi Model VE 50 GD or equivalent.
- b. Flask round bottom, 250 ml, with standard taper neck to fit evaporator.
 - c. Water aspirator glass or polyethylene.
- 4.5.7.3 Non-Volatile Residue. This method involves the gradual evaporation of hydrazine sample on an explosion proof hot plate in a fume hood. The difference in weight before and after evaporation is calculated as non-volatile residue (NVR).

4.5.7.3.1 Equipment.

CAUTION

Metal surfaces such as iron, platinum, etc., may reduce the ignition temperature of hydrazine, therefore, care must be taken to ensure that Pyrex is always used in this equipment.

- a. Hot plate, explosion proof.
- b. High efficiency fume hood.
- 4.5.7.3.2 <u>Sample Analysis</u>. Place three clean 125-ml Erlenmeyer flasks in an oven at 100°C for 30 minutes. Label one as the blank and the others as Samples 1 and 2. Remove flasks from oven and place in a desiccator to cool for one hour. Weigh each clean, dry flask to the nearest 0.0001 g. Record the sample flask weight as A and the blank flask weight as C. Pipet 40 ml of hydrazine sample into the sample flasks marked 1 and 2. Add 40 ml of water to each sample of hydrazine in flasks 1 and 2. Hydrazine and water form an azeotrope (constant boiling mixture) with a maximum boiling point of 120.5°C

(771 mm Hg pressure). Therefore, when anhydrous hydrazine and water are mixed, a corresponding eutectic of 76.0 mole percent hydrazine is the maximum concentration obtained. This eliminates any significant hazard in evaporating a hydrated sample of hydrazine to dryness. Pipet the same volume of distilled water into the blank flask as was pipetted into the sample flask. Place each flask on a hot plate which is set to a temperature 150-200°C. Evaporate to dryness, but do not bake. Remove flasks from hot plate and place them in oven set at 110°C for 30 minutes. Remove flasks from oven and place in a desiccator for one hour to cool. Weigh each flask to the nearest 0.0001 g. Record the sample flask weight as B and the blank flask weight as D.

NOTE

Save the residue for chloride determination (4.5.4).

4.5.7.3.3 Calculations.

%NVR =
$$\frac{\text{(Weight B - Weight A) - (Weight D - Weight C) x 100}}{\text{Sample Size, ml x density of N2H4, g/ml}}$$

WHERE:

NVR	=	Non-volatile residue	
Α	=	Weight of sample flask	
В	z	Weight of sample flask + residue	
С	=	Weight of blank flask	
D	=	Weight of blank + H ₂ O residue	

- 4.5.8 <u>Carbon Dioxide</u>. The carbon dioxide content of hydrazine shall be determined by one of the following methods. Method 4.5.8.1 shall be the referee method.
 - 4.5.8.1 Determination of carbon dioxide by Gas Chromatographic analysis.
- 4.5.8.1.1 Procedure. Assemble the apparatus as shown in Figure 2 and connect a gas buret to the gas exit. Adjust the needle valve so that the flow at the gas exit is 50-100 ml/min. Adjust the gas chromatograph carrier gas flow to 30-60 ml/min, the column temperature to 100° to 150° C, and the detector temperature to 150° to 200° C. Adjust the detector current to the manufacturer's recommended level for nominal sensitivity. Position the gas sampling valve and the stopcocks so that the sparge gas passes through the $C0_2$ trap and allow the system to purge in this manner for 45 min. After the apparatus has purged, cool the $C0_2$ trap to liquid nitrogen temperature to collect a sample for background $C0_2$ determination. Collect the sample for 30 to 45 min and then position the stopcocks so that the sparge gas bypasses the

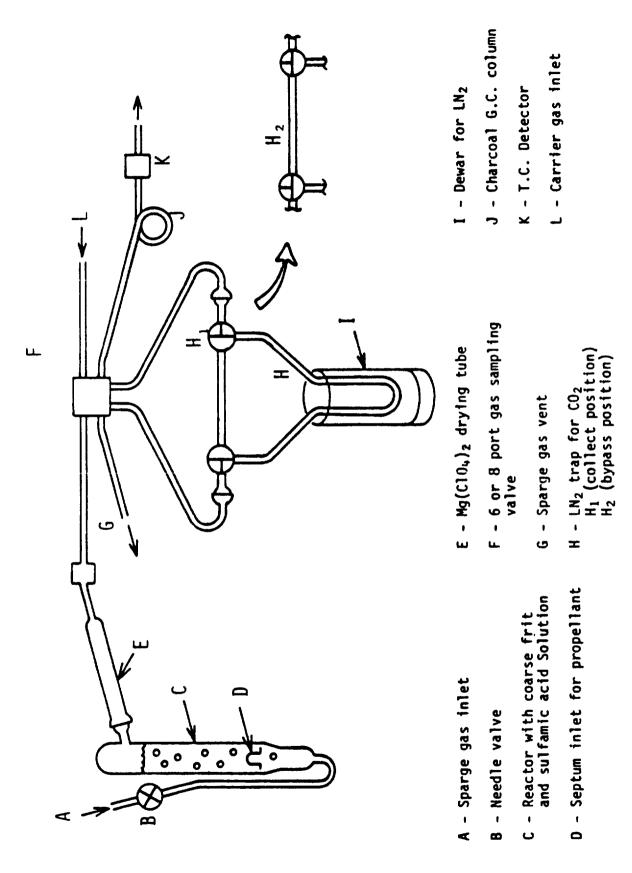


FIGURE 2, CO2 APPARATUS

cooled portion of the trap. Position the gas sampling valve so that the carrier gas will flow through the bypass line of the trap, and warm the lower portion to ambient temperature. Inject the trapped gases into the gas chromatograph by rotation of the stopcocks to the collect position. Determine the CO_2 peak height or area and record the results. Position the gas sampling valve so the sparge gas is again flowing through the trap and cool the trap to liquid nitrogen temperature. Immediately inject a 0.25 ml propellant sample into the septum inlet of the apparatus and allow 30 to 45 min for complete transfer of the CO_2 from the sulfamic acid bubbler to the trap. Repeat the trapped sample injection sequence described earlier for the background determination and record the sample CO_2 peak height or area.

PROPELLANT TRANSFER. To achieve accurate CO_2 results, all propellant transfers must be performed in a CO_2 -free atmosphere (i.e. a gaseous nitrogen filled dry box). Propellant in the sampler shall be transferred in an inert atmosphere to septum sealed vials. The vials shall be stored in an inert gas purged container when not actually being sampled. Use an ordinary 1/2 cc glass syringe with a small amount of "Apiezon L". The gas tight syringe shall be cleaned and dried between each propellant sample injection into the apparatus.

4.5.8.1.2 <u>Calculation</u>. Calculate the CO_2 level of the propellant as follows:

$$CO_2 = (R_S - R_b) \times S$$

WHERE:

 $(R_S - R_D)$ = CO_2 response due to the sample minus CO_2 response due to the background, peak height or area.

S = Sensitivity, %CO₂ per unit response.

4.5.8.1.3 Calibration. Accurately weigh 0.130 g ammonium carbonate monohydrate (ACS) $(\overline{NH}_{u_1})_2\overline{CO}_3H_2\overline{O}$ into a tared glass vessel which will accept a vapor tight cap and will contain 100 ml of liquid. Place the vessel in the propellant transfer apparatus (dry box) which contains the propellant sample. Transfer 100 ml of propellant to the vessel, cap the vessel. Use sample or other known hydrazine. Dissolve and thoroughly mix the ammonium carbonate solution. Remove the vessel from the transfer apparatus and weigh the container. Calculate the CO_2 added as follows:

$$%CO_2$$
 added =
$$\frac{0.130 \times 44.01 \times 100}{(\text{tot. wt. - [tare + 0.130]}) \times 114.1}$$

Transfer a portion of the calibration standard to a septum sealed vial and use the procedure described in 4.5.8.1 to determine the response due to the CO_2 sensitivity of the apparatus as follows:

$$S = \frac{\text{%CO}_2 \text{ added}}{R_{\text{std}} - R_{\text{s}}}$$

WHERE:

 $R_{std} - R_s$ = The response due to the CO_2 in the standard minus the response due to the sample, peak height or area.

4.5.8.1.4 Reagents and Equipment.

4.5.8.1.4.1 Reagents.

- a. Sulfamic Acid. aqueous, 90% saturated solution.
- b. Bromcresol Green, indicator, several drops added to the sulfamic acid solution will provide a visual indication of when the solution is spent.
- c. Charcoal, coconut, 60/80 mesh, activated, or column of equivalent resolution.

4.5.8.1.4.2 Equipment.

- a. Reactor, sulfamic acid, constructed so that the volume is approximately 75 cc, Borosilicate glass, See Figure 2.
- b. Drying tube, $Mg(C10_4)_2$, approximately 6" x 1/2", Borosilicate glass.
- c. Trap, CO_2 , Borosilicate glass, constructed with 6 mm 0.D. tubing, the lower loop should contain glass beads to increase the condensing surface area.
 - d. Valve, gas sampling, 6 or 8 port.
 - e. Valve, needle, for regulation of the sparge gas.
 - f. Dewar, conical, to fit trap.
- g. Gas chromatograph, equipped with a thermal conductivity detector, $6' \times 1/8'' 60/80$ mesh charcoal column.
 - h. Recorder, potentiometric, 1 mv span, 1 sec. F.S. pen speed.
 - 1. Integrator, mechanical or digital, optional.
- 4.5.8.2 Determination of carbon dioxide in hydrazine propellants by infrared analysis. This method is to be utilized to determine concentration

of carbon dioxide (CO_2) in propellant hydrazines, and monopropellant within a range of 1-2 mg/kg (ppm). The sample of propellant is injected into a solution of 20% phosphoric acid which absorbs the hydrazine and ammonia. Carbon dioxide is liberated from the acidic solution and is swept out of the solution by a stream of nitrogen through a sulfuric acid solution to remove water vapor and measured by infrared technique.

4.5.8.2.1 Calibration Procedure.

- a. Assemble the apparatus as shown in Figure 3.
- b. Adjust the flow of nitrogen purge gas to 200 cc/min by use of flowmeter.
- c. Add 200 ml of 20% phosphoric acid containing several drops of Bromcresol Green indicator to the CO_2 reaction flask, turn on the magnetic stirrer to give vigorous, constant stirring. Once set, the stirring rate and nitrogen flow should be kept constant throughout the analysis.
- d. After the apparatus has purged for 30 minutes, set the power to RANGE 1 position. Adjust the zero on the infrared analyzer by using one zero adjusting knob and the shutter setting.

4.5.8.2.2 Standardization.

- a. Inject 25 microliters of standard anhydrous sodium bicarbonate solution into the phosphoric acid (25 ppm level) solution via the septum on the phosphoric acid containing unit.
- b. Adjust the gain control so the 25 mg/Kg standard will read 50% of the scale on the infrared analyzer or the strip chart recorder (it takes two minutes to reach maximum deflection from the infrared analyzer. 1 scale unit = 0.5 mg/Kg $\rm CO_2$). Wait ten minutes or until the infrared analyzer scale goes back to zero.
- c. Repeat with an injection of 10 microliters scale on infrared analyzer should read 20 units (10 mg/Kg $\rm CO_2$).
- 4.5.8.2.3 <u>Sample Analysis</u>. Inject 1 ml of hydrazine sample into the phosphoric acid reaction flask. Record the reading from the infrared analyzer scale or the strip chart recorder (from the zero point).
- 4.5.8.2.4 Reagents. Accurately weigh 0.19 g of anhydrous sodium bicarbonate (NaHCO $_3$) reagent grade into 100-ml volumetric flask and dilute to volume with water. A 25 ml sample of this solution will provide .25 mg of CO $_2$ per ml of the phosphoric acid solution.

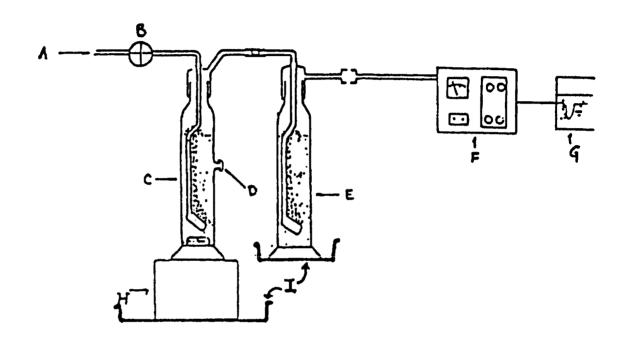
4.5.8.2.5 <u>Equipment</u>.

a. Reactor, phosphoric acid, 250-ml glass wash bottle.

Mil-P-26536D

FIGURE 3

CO₂ APPARATUS



- A. Sparge gas inlet
- B. Needle valve
- C. Reactor and phosphoric acid solution
- D. Septum inlet for propellant

- E. Conc. H₂SO₄ trap for removing H₂O
- F. Infrared Analyzer
- G. Recorder
- H. Magnetic stirrer
- I. Plastic Spill Trays

- b. Drying bottle, 200-ml glass wash bottle filled with concentrated sulfuric acid solution.
 - c. Beckman Model 865 non-dispersive infrared analyzer.
- 4.5.9 Other Volatile Carbonaceous Material. Other volatile carbonaceous material shall be determined by a Gas Chromatographic Method.
- 4.5.9.1 Procedure. Use the column, test conditions, and sample size described in para 4.5.2.1. A flame ionization detector shall be used. UDMH, methanol, and isopropyl alcohol emerge from the column as a single peak and MMH emerges immediately after the water peak. Ammonia, which emerges prior to the combined UDMH/Alcohols peak shall not be summed with the carbonaceous material. Aniline will emerge from the column 30-45 minutes after the first injection. Allow the baseline to stabilize before injecting a second series of samples.
- 4.5.9.2 <u>Calibration</u>. Add 9.4 microliters of UDMH and 8.3 microliters of MMH to 75 milliliters of propellant sample contained in a 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 UDMH and MMH peaks.
- 4.5.9.3 Calculations. The combined UDMH/Alcohols peak shall be calculated as percent UDMH. MMH is calculated separately.

$$\%UDMH = \frac{0.01 A_S}{A_C - A_S}$$

WHERE:

As = Area of sample UDMH times its attenuation factor.

A_C = Area of calibration UDMH times its attenuation factor.

%MMH =
$$\frac{0.01 A_S}{A_C - A_S}$$

WHERE:

 A_{S} = Area of sample MMH times its attenuation factor.

A_C = Area of calibration MMH times its attenuation factor.

% Other volatile carbonaceous material = %UDMH + %MMH.

NOTE

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

4.5.9.4 Reagents and Equipment. The following reagents and equipment shall apply as test conditions of 4.5.9.

4.5.9.4.1 Reagents.

- a. UDMH conforming to MIL-P-25604.
- b. MMH conforming to MIL-P-27404.
- 4.5.9.4.2 Equipment. As required in 4.5.2.1, except gas chromatograph shall be equipped with a flame ionization detector, splitless capillary inlets.
- 4.6 <u>Preparation for Delivery Inspection</u>. 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 100-199, or DOT exemption obtained by the shipper in accordance with DOT 49 CFR 107.101.
- 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 Type 304 or 347 stainless steel or 42B, 42C, or 42D 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 310, MC 311, or MC 312 of Types 304L or 347 stainless steel with molybdenum content not exceeding one-half of one percent.
- 5.1.3 Approved Tank Cars. Tank cars which are approved by DOT for this propellant shall conform to DOT specifications 103CW or 111A100W6 of Types 304L or 347 stainless steel with molybdenum content not exceeding one-half of one percent, or 103AALW of aluminum. 111A100W6 tank cars shall not be equipped with bottom outlets.
- 5.1.4 Other Containers. Other containers of any type which does not presently have DOT approval shall conform to the requirements of DOT special permit.

- 5.2 <u>Preparation of Containers</u>. 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 is found of internal contamination, 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 <u>Gaskets</u>. 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 1 Grade B, at not less than atmospheric pressure.
- 5.4 <u>Labeling and Marking</u>. 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. The product meets the criteria of DOT CFR 49 para 173.3a(b)(2). Containers shall be marked "INHALATION HAZARD."
- 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. ANSI Z129.1 should be used as a guide in preparation of the precautionary label to be applied to each drum.
- 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 "Hydrazine." Any other name identification shall be obliterated by removing or overpainting.

NOTES

- 6.1 Intended Use. The propellant covered by this specification is intended for use as a fuel in rocket engines.
 - 6.2 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. Grade of propellant ordered.
- 6.3 Highway Safety. To promote safety in the transportation of propellants in interstate commerce by motor vehicle, the shipper should assure that each driver possesses Emergency Response Guide Book, DOT P 5800.3.
- 6.4 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(s) 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.
- 6.5 Subject Term (Key Word) Listing:

Hydrazine

Monopropellant

Rocket fuel

Custodian

Preparing Activity:

Air Force - 68

Army - MI

Navy - AS

Air Force - 68

Review Activities:

NASA

Army - MI

Navv - AS

Air Force - 12, 19

Project No. 9135-0118

Civil Agency Interest:

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