

MIL-P-87930 (USAF)
25 October 1977

MILITARY SPECIFICATION
PROPELLANT, HYDRAZINE-WATER
(70% Hydrazine - 30% Water)

This specification is approved for use by the Air Force Rocket Propulsion Laboratory (LKCP), Department of the Air Force, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 This specification covers the requirements for a hydrazine-water propellant blend, 70 percent by weight hydrazine and 30 percent by weight water.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

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 suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

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

FSC 9135

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DEPARTMENT OF TRANSPORTATION

49 CFR 170-190

Hazardous Materials Regulations
of the Department of Transportation

(Applications for copies should be addressed to the Superintendent of Documents,
Government Printing Office, Washington, DC 20402.)

MANUFACTURING CHEMISTS' ASSOCIATION, INC.

Manual L-1

A Guide for the Preparation of
Warning Labels for Hazardous Chemicals

(Applications for copies should be addressed to the Manufacturing Chemists'
Association, 1825 Connecticut Avenue, N. W., Washington, DC 20009.)

AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM D 2276-73

Tests for Particulate Contamination in
Aviation Turbine Fuels

ASTM D 1068-68

Tests for Iron in Water and Waste Water

ASTM E 29-73

Recommended Practice for Indicating which
Places of Figures Are to be Considered
Significant in Specified Limiting Values.

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

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.

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, unless otherwise specified (6.2).

TABLE I, CHEMICAL AND PHYSICAL PROPERTIES

PROPERTIES	LIMITS	TEST PARAGRAPH
Hydrazine (percent by weight) - max - min	70 69	4.5.2
Water (percent by weight) - min	30	4.5.2
Particulate (Milligrams per liter)	1.0 max	4.5.3
Chloride (percent by weight)	0.0005 max	4.5.4
Aniline (percent by wieght)	0.40 Max	4.5.5

PROPERTIES	LIMITS	TEST PARAGRAPH
Iron (percent by weight)	0.002 max	4.5.6
Nonvolatile Residue (percent by weight)	0.004 max	4.5.7
Carbon Dioxide (percent by weight)	0.002 max	4.5.8
Other Volatile Carbonaceous Material (Total as either MMH or UDMH/Alcohol, percent by weight)	0.02 max	4.5.9

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 righthand 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-29-67).

3.3 Filter. A filter with a 2-micron nominal and 10 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 inspections 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 (6.2).

4.2 Classification of tests. 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 test 4.4.1
- b. Sampling tests 4.4.2

4.4.1 Individual test. Each container of propellant shall be sampled and each resulting sample shall be subjected to the following test:

- Examination of product 4.5.1

4.4.2 Sampling tests. 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. For containers of 100 gallons or less water capacity, 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 is used to fill shipping containers directly from the process output. A continuous input of raw materials and output of finished product by one manufacturer in one plant with no 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 Sample. A sample consists of not less than 1500 milliliters (ml) of propellant. Unless otherwise specified, quality conformance tests shall be made on the sample of the 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 bottles intended for sampling shall be specially cleaned and handled in accordance with procedure described in ASTM 2276-73 with the following addition: The sample container must be dried with filtered nitrogen after cleaning. 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 to cover the container purged with nitrogen is strongly recommended.

4.4.2.1.3 Drums. 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.

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TABLE II. SAMPLING FOR TEST

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

4.4.2.1.4 Portable Tanks, cargo tanks, and tank cars. 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 fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected (6.2).

4.5 Test methods.

4.5.1 Examination of product. The propellant shall be examined to determine compliance with the requirement specified in 3.4. The propellant may be visually examined while performing test specified in 4.5.3.

4.5.2 Hydrazine, water, and ammonia. The propellant, water, and ammonia content of the sample shall be determined by the following method.

4.5.2.1 Suggested column preparation. Weigh 2 grams of polyethylene glycol 400 and 18 grams of 60/80 mesh Fluoropak support material into separate beakers. Dissolve the polyethylene glycol 400 in reagent grade methylene chloride (dichloromethane). The final volume of the solution should be approximately that of the support material. Pour the support material into the polyethylene glycol 400 (stationary phase) solution with gentle stirring. Evaporate the solvent by spreading the mixture in a tray. Occasionally turn the mixture gently during the drying process. The column packing material is dry when it becomes a free-flowing powder. Fill a 1/8 inch x 2 feet stainless steel tube by pouring the prepared material through

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a small funnel attached to one end. The bottom of the tubing is plugged with a small wad of glass wool or capped. Use a mechanical vibrator to facilitate packing. When the column is full, plug the ends with glass wool and bend the tubing to the configuration required by the chromatograph oven.

4.5.2.2 PROCEDURE Install the prepared column into the gas chromatograph but do not connect the column to the detector inlet. Condition the column for at least 4 hours by heating at 100°C with the carrier gas (helium) flow set at approximately 20 milliliters per minute. After conditioning the column, set the column oven temperature at 100°C and connect the column to the detector inlet. Adjust the carrier gas flow to 20-50 milliliters per minute. If the gas chromatograph is equipped with separate injector and detector temperature controls, set the detector at 150°C and the injector at 110°C. Saturate the column by injecting two or three 5 microliter samples of propellant. Saturate the column prior to each series of analyses. When more than 30 minutes elapse between the elution of the hydrazine and the injection of a new sample, resaturate the column with one 5-microliter injection of propellant sample. Use a clean, dry 10-microliter hypodermic syringe and draw up 8 microliters of sample. Invert or pump the syringe to expel the gas bubbles. Carefully set the syringe plunger to the 1-microliter mark and wipe the tip with a piece of tissue without touching the open end of the needle. Quickly inject the sample into the instrument injection port and then withdraw the syringe immediately. Measure the areas of all peaks in the chromatogram. The analyst may vary the temperature, flow rate, column size, and sample size to optimize the analysis.

4.5.2.3 CALCULATIONS.

$$\%N_2H_4 = \frac{A_{HKH}}{\sum A_i K_i (100 - \% \text{ Aniline})} \times 100$$

$$\%H_2O = \frac{A_{WKW}}{A_1 K_1 (100 - \% \text{ Aniline})} \times 100$$

$$\%NH_3 = \frac{A_A K_A}{\sum A_i K_i (100 - \% \text{ Aniline})} \times 100$$

where: A_{HKH} , A_{WKW} , $A_A K_A$ equals the areas of the hydrazine, water, and ammonia peaks multiplied by their respective sensitivity factors.

$\sum A_i K_i \left(\frac{100}{100 - \% \text{ Aniline}} \right)$ equals the sum of

area/sensitivity factor products of all peaks in the chromatogram corrected for the aniline content of the propellant.

Note: Assume $K_H = K_A = 1.000$

4.5.2.1.4 CALIBRATION. Determine the hydrazine and water content of a sample of anhydrous hydrazine using the procedure described in 4.5.2.1.2 and calculate the results as shown in 4.5.2.1.3. Assume $K_H = K_W = K_A = 1.000$. Using at least 10g of this analyzed hydrazine, prepare a mixture with distilled water which is approximately 70% hydrazine and 30% water. Use an analytical balance that is accurate to at least ± 1 mg. Calculate actual composition of the mixture as follows:

$$\%N_2H_4 = \frac{\text{wt. } N_2H_4 \times \%N_2H_4 \text{ (assay)}}{\text{Total wt.}}$$

$$\%H_2O = \frac{\text{wt. } H_2O + \left(\frac{\text{wt. } N_2H_4 \times \%H_2O \text{ (assay)}}{100} \right)}{\text{Total wt.}} \times 100$$

where: $\%N_2H_4$ (assay), $\%H_2O$ (assay) equals the hydrazine and water content of the anhydrous hydrazine used

wt. N_2H_4 , wt H_2O = the weights of anhydrous hydrazine and water to prepare the mixture. Total wt. equals wt. N_2H_4 + wt. H_2O .

$$K_W = \frac{A_H \% H_2O}{A_W \% N_2H_4}$$

Use K_W as determined above in 4.5.2.1.3 for the propellant sample.

4.5.2.1.5 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.

(3) Tubing stainless steel, 1/8 inch O.D. x 2 feet.

(4) Syringe: 10ml capacity.

b. Reagents

(1) Dichloromethane ACS Grade.

(2) Polyethylene glycol 400 Carbowax 400, or equivalent

(3) Fluoropak 80: 60/80 mesh, Analabs, Inc. 9 Hobson Avenue,

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Hamden, Ct 06518, or equivalent.

(4) Hydrazine anhydrous conforming to Mil-P-26536C, Amendment 1

(5) Helium conforming to Mil-P-27407.

4.5.3 Particulate. The propellant sample shall be tested for contamination in accordance with ASTM Designation D-2276-73, Method A, with the following exceptions.

4.5.3.1 Mix the sample thoroughly without exposure to air. Immediately pour 100 ml of the sample into a clean 1000-ml graduated cylinder. Use this 1000 ml of propellant for the particulate analysis.

4.5.3.2 Use a solvent resistant filter disc made from such materials as Millipore FALP04700 or Gelman VF-6, (Fluoride- Metrical), plain, white, 10 + 3 microns, 47 mm diameter or equivalent for monopropellant grade instead of that specified in Method 2276-73

4.5.3.3 The drying oven temperature shall be 158°F (70°C) instead of the 194°F (90°C) specified in Method 2276-73.

4.5.3.4 Filtered isopropyl alcohol shall be used for rinsing the sample bottle and filter holder instead of petroleum ether specified in Method 2276-73. Wet the filter with filtered isopropyl alcohol prior to filtering the sample.

4.5.4 Chloride. The chloride content of the propellant shall be determined by the following 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-H₂SO₄. 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 the 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 2 to 3 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 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.

4.5.4.2 Calibration. Weigh exactly 2.103g of KCl and transfer to a 1000 ml volumetric flask. Dissolve the salt in distilled water and dilute to the mark. Mix the solution thoroughly and transfer to a 1000 ml plastic bottle. This solution contains 1000 ppm Cl. To each of four 1000 ml volumetric flasks add 400 ml of 0.1 N-H₂SO₄ solution. Prepare dilute chloride standards by pipeting 5.00, 10.0, 25.0, and 50.0 ml of the 1000 ppm chloride standard sequentially into the four flasks. Add 2-3 drops of the bromcresol green indicator to each flask and titrate with 1 N-NaOH to the green endpoint. Dilute

each to the mark with distilled water, mix thoroughly, and transfer the standards to 1000 ml plastic bottles. The chloride levels of these standards are 5, 10, 25, and 50 ppm, respectively. Pour approximately 10 ml of each standard into one of four 25 ml beakers and starting with the 5 ppm chloride solution, immerse the electrodes and record the resulting millivolt reading. Rinse the electrode with distilled water and wipe with a clean tissue between each standard and each sample.

4.5.4.3 Calculation. Calculate the percent chloride in the sample using the following formula.

$$\%Cl^- = \frac{\text{ppm } Cl^- (\text{From curve}) \cdot (25)}{1.03 \times 10^6}$$

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-H₂SO₄ = Pour 3 ml of ACS grade concentrated H₂SO₄ 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 chloride-free distilled water, and mix thoroughly. Transfer the solution to a plastic bottle.

c. 1 N-NaOH - Dissolve 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.

d. Potassium Chloride - KCl, ACS grade, dried for 4 hours at 100°C.

e. Bromcresol Green indicator - 1% aqueous solution. Use chloride-free distilled water.

f. Silver nitrate = ACS grade.

4.5.4.4.2 Equipment.

a. Analytical Balance = 100 g capacity, 0.1 mg sensitivity.

b. Volumetric Flasks = 1000 ml and 25 ml capacity.

c. Volumetric Pipets = 5.00, 10.0, 25.0, and 50.0 ml capacity.

d. Eye Droppers

e. Beakers = 25 ml capacity, plastic, disposable.

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- f. Bottles = 1000 ml capacity, plastic, narrow mouth, screw cap.
- g. Chloride Electrode - solid state Orion Model 94-17, or equivalent.
- h. Reference Electrode, Double Junction, Orion Model 90-02 with 1M-KNO₃ outer compartment filling solution, or equivalent.
- i. pH/Millivolt meter - Orion Ionanalyzer 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.

4.5.5.1 Spectrophotometric Method.

4.5.5.1.1 Procedure. Pipet or syringe 0.25 ml (250 μ l) 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 path length 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 μ l) 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 these 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 calibration standards should be prepared fresh daily.

4.5.5.1.3 Reagents and Equipment. The following shall apply as test conditions of 4.5.5.1.

4.5.5.1.3.1 Reagents.

- a. Distilled Water
- b. Aniline, ACS grade

4.5.5.1.3.2 Equipment.

- a. Syringes = 0.05 ml (50 μ l) and 0.5 ml (500 μ l) capacities.
- b. Pipets = Volumetric, 2.0, 4.0, 6.0, and 8.0 ml capacities or one 10 ml syringe graduated in 0.2 ml increments.
- c. Volumetric flasks, 100 ml and 25 ml capacities.
- d. Spectrophotometer, double beam, ultraviolet, 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 column as described in 4.5.2 except substitute a mixture consisting of 0.5 g of Amine 220 and 9.5 g of Apiezon L for the propylene glycol 400. Also use a white, silanized 60/80 mesh diatomaceous earth support instead of Anakrom B. Condition the column for 4 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 μ l 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 Calibration. Pipet or syringe 0.5ml (500 μ l) of aniline into a 100 ml volumetric flask containing approximately 50 ml of distilled water, shake the mixture thoroughly. This calibration standard is equivalent to 0.5% aniline in propellant. Inject 0.5 μ l 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 Calculation. Use the following formula for calculation of the aniline content of the propellant.

$$\% \text{ aniline} = \frac{0.5 A_s}{A_c}$$

Where:

A_s = 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 Reagents and Equipment. The following reagents and equipment shall apply as test conditions of 4.5.5.2.

4.5.5.2.4.1 Reagents.

- a. Aniline - ACS grade
- b. Distilled water

4.5.5.2 4.2 Equipment.

- a. Volumetric flask = 100 ml capacity
- b. Syringe = 1.0 ml capacity
- c. Pipet = 0.5 ml capacity or syringe 0.5 ml (500 μ l) capacity
- d. Gas chromatograph, equipped with a flame ionization detector.
- e. Recorder, potentiometric, with integrator, pen speed = 1 sec. F.S., 1 mv span.

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 Method.

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-H₂SO₄ to the vessel and place on a hot-plate stirrer. Heat the mixture, while stirring, to boiling and maintain at that temperature for 3 to 5 minutes. Allow the solution to cool and transfer to a 200 ml volumetric flask. Rinse the vessel at least three times with 5 ml portions of 0.1 N-H₂SO₄ combining all of the rinses in the volumetric flask. Dilute to the mark with distilled water and mix thoroughly.

Determine the absorbance of the sample at an iron wavelength closest to 2480 Å on suitable atomic absorption spectrophotometer, after setting the instrument to zero absorbance, with 0.1 N-H₂SO₄. Compare the results to a calibration curve prepared from iron standards and record the iron-concentration of the sample.

4.5.6.1.2 Calibration. 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 each 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 Calculation. Calculate the iron content of the propellant using the following formula.

$$\% \text{ Fe} = \frac{(\text{ppm Fe})}{1.03 \times 10^4}$$

Where. ppm Fe = the iron content of the sample solution, using the calibration curve.

4.5.6.1.4 Reagents and Equipment. The following reagents and equipment shall apply as test conditions of 4.5.6.1.

4.5.6.1.4.1 Reagents.

- a. Iron = ACS grade, wire or shot.
- b. 5 N-H₂SO₄ = carefully pour 180 ml of concentrated ACS grade, H₂SO₄ into 600 ml of distilled water contained in a 1000 ml volumetric flask, cool the solution, dilute to the mark with distilled water, and mix thoroughly.
- c. 0.1 N-H₂SO₄ = prepare as above except use 3 ml of concentrated ACS grade H₂SO₄.
- d. Distilled water.

4.5.6.1.4.2 Equipment.

- a. Volumetric flasks = 1000 ml and 200 ml capacities.
- b. Volumetric pipets = 1.0, 2.0, 3.0, and 5.0 ml capacities.
- c. Hotplate/stirrer = controllable heat and stir settings.
- d. Atomic Absorption Spectrophotometer, Perkin Elmer 290, 303, or equivalent with an iron hollow cathode lamp.

4.5.6.2 Colorimetric Method.

4.5.6.2.1 Procedure. 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 Non-Volatile Residue. 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 Hg. 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

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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 syringe equipped with a polytetrafluoroethylene needle as shown. With the nitrogen purge in place, turn on the aspirator and reduce the apparatus pressure to less than 30 mm Hg. 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 2-4 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 Calculations. Calculate the non-volatile residue of the propellant as follows:

$$\% \text{ NVR} = \frac{W_1 - W_0}{1.03} \quad \text{where:} \quad \begin{array}{l} W_0 = \text{Wt. clean vessel} \\ W_1 = \text{Wt. residue + vessel} \end{array}$$

Report the averaged result of duplicate NVR determinations.

$$\text{Average \% NVR} = \frac{(\% \text{ NVR})_1 + (\% \text{ NVR})_2}{2}$$

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

4.5.7.1.3.1 Reagents.

- a. Distilled water
- b. Gaseous Nitrogen = conforming to Mil-P-27401, or equivalent

4.5.7.1.3.2 Equipment. (See Figure 1)

- a. Balance = Analytical, 100 g capacity, 0.1 mg sensitivity.
- b. Beaker-glass, 1000 ml capacity
- c. Tissue - paper, lintless
- d. Syringe - glass, 100 ml capacity, equipped with a 8-12 inch polytetrafluoroethylene needle.

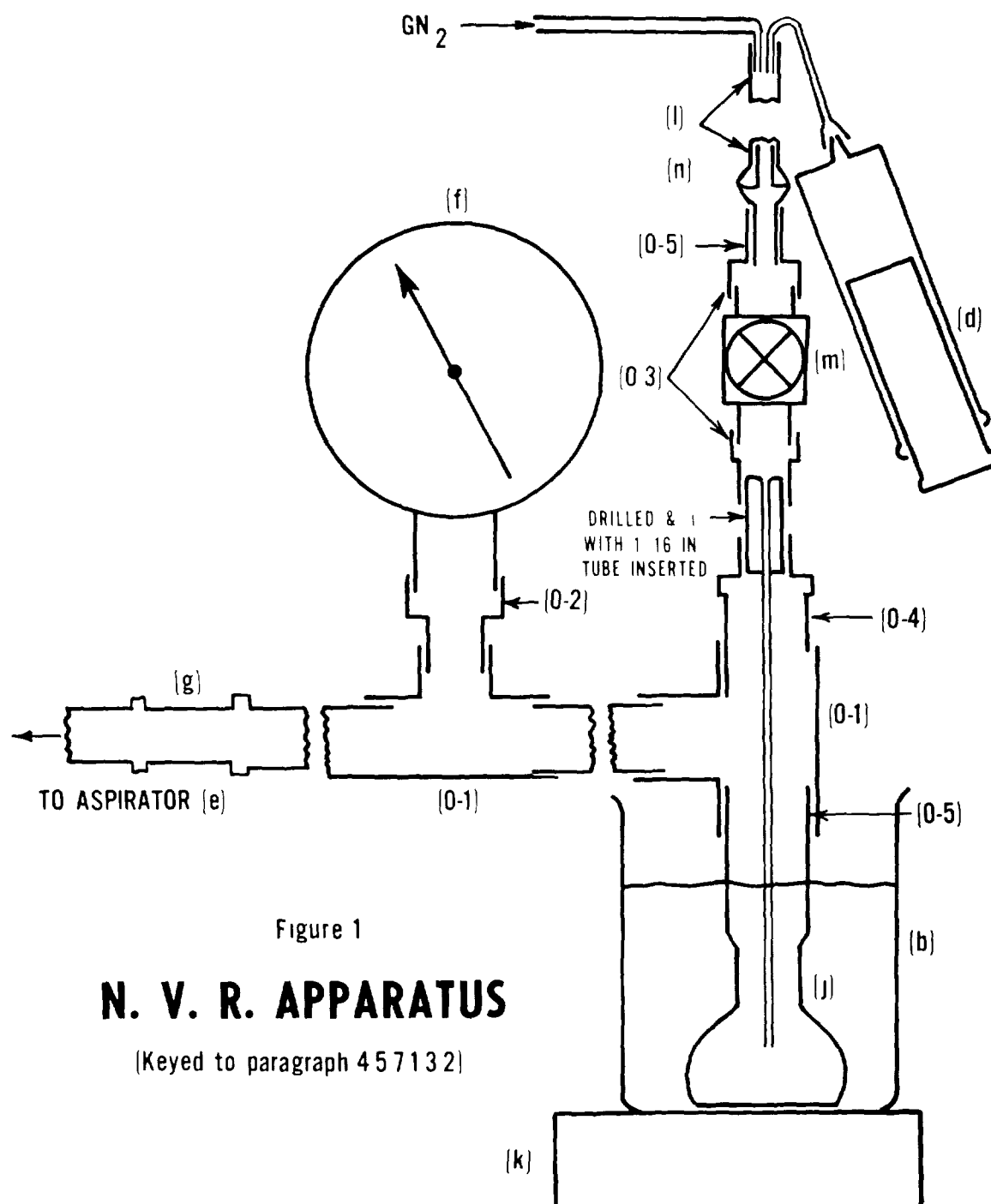


Figure 1

N. V. R. APPARATUS

(Keyed to paragraph 4 5 7 1 3 2)

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- e. Water aspirator = glass or polyethylene
- f. Gauge = Absolute pressure, 0-760 mm range, stainless steel, Matheson Gas Co. Model No. 63-5601, or equivalent.
- g. Connector = Polyethylene, separable, Sargent Cat No. 73788, or equivalent.
- h. Tubing = Polyethylene, 1/2 inch O.D., borosilicate glass, 1/2 inch O.D., 1/4 inch O.D., stainless steel, 1/16 inch O.D. x 0.025 inch wall.
- i. Rod = polytetrafluoroethylene or polyethylene, 1/4 inch O.D.
- j. Flask - volumetric, borosilicate glass, 25 ml capacity.
- k. Hotplate = adjustable heat range
- l. Pipet = Volumetric, borosilicate glass, 100 ml capacity.
- m. Valve = stainless steel, polytetrafluoroethylene packing, Whitey Cat. No. SS-ORM2, or equivalent.
- n. Joints = borosilicate glass, spherical, 12/5, ball, socket, and clamp.
- o. Fittings = Stainless steel, Swagelok or equivalent as follows:
 - (1) Union tee, 316-810-3 (2 each)
 - (2) Adapter, 316-811-A-4F
 - (3) Connector, 316-400-7-2 (2 each)
 - (4) Reducer, 316-400-R-8
 - (5) Ferrules, T-814-1, T-813-1, T-4-4-1, T-403-1
- p. Clamps = hose, to fit 1/2 inch O.D. tube.

4.5.7.2 Rotary Evaporation Method

4.5.7.2.1 Procedure. Dry a 250 ml round bottom flask in an oven at 100°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 propellant 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 (1) liter suction flask and evaporate the propellant to dryness. A warm bath may be placed around the rotating flask to aid evaporation.

Caution: Do not exceed 40°C. Safety glasses must be worn throughout the entire evaporation process.

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When no more hydrazine distills over, the water bath is lowered. Remaining water and high boiling components (mainly aniline) are distilled under vacuum 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 (1) hour and weighed as before to a constant weight.

Caution: The contact of hydrazine vapor with metal surfaces, as in a metal rotary evaporator, must be avoided.

4.5.7.2.2 Calculations.

$$\% \text{ Non-Volatile Residue} = \frac{\text{Weight of Residue, g} \times 100}{V \times D}$$

V = Volume of Sample, ml

D = Density of propellant at Measured Temperature

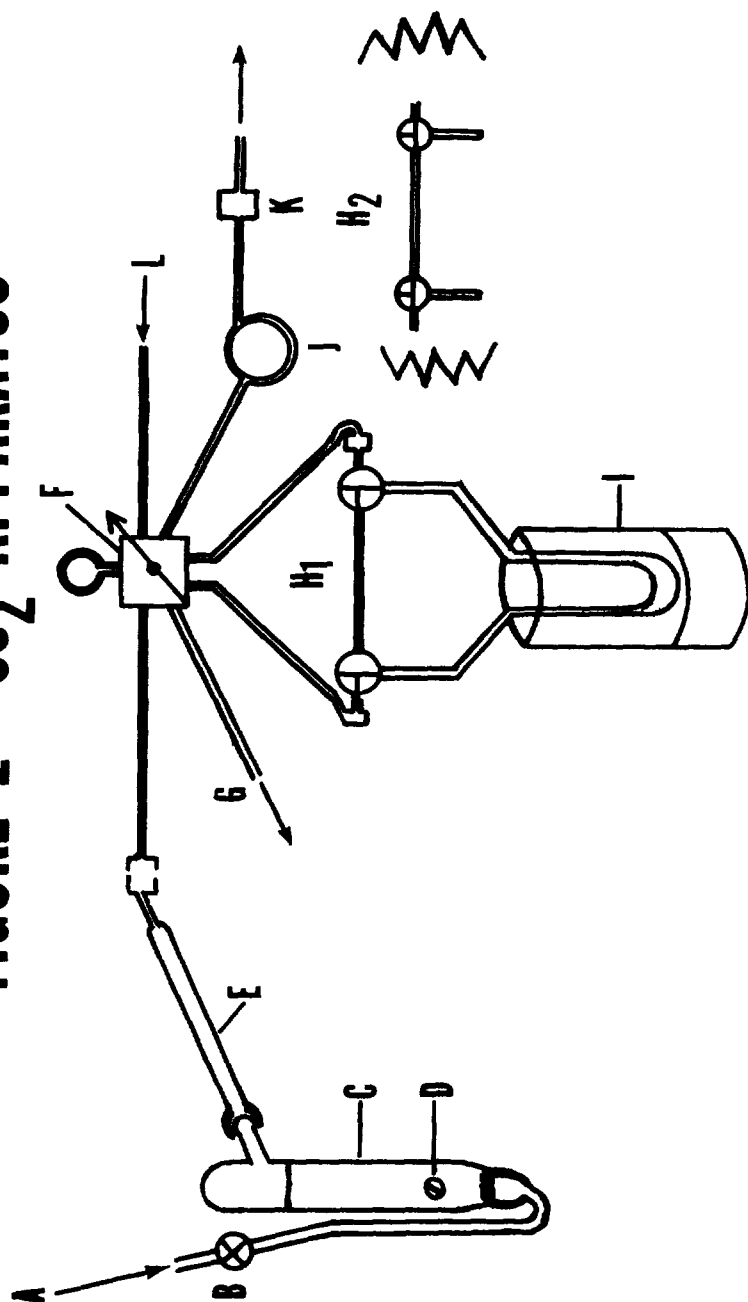
4.5.7.2.3 Equipment.

- a. All glass rotary evaporator, Rinco-Buchi Model VE 50 GQ or equivalent.
- b. Flask round bottom, 250 ml, with standard taper neck to fit evaporator.
- c. Desiccator
- d. Water Aspirator
- e. Water Bath (40°C)
- f. Hot Air Gun
- g. Drying Oven (110°C)
- h. Analytical Balance
- i. Standard Taper Teflon Sleeve
- j. Safety Glasses

4.5.8 Carbon Dioxide. The carbon dioxide content of the propellant shall be determined by the following method.

4.5.8.1 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 50-100ml/min. Adjust the gas chromatograph carrier gas flow to 30-60ml/min, the column temperature to 100° to 150°C, and the detector temperature to 150°-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 CO₂ trap and allow the system to purge in this manner for 45 min. After the apparatus has purged, cool the CO₂ trap to liquid nitrogen temperature to collect a

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FIGURE 2 CO₂ APPARATUS

- | | | |
|---|--|---------------------------------|
| A - SPARGE GAS INLET | E - $Mg(ClO_4)_2$ DRYING TUBE | I - DEWAR FOR LM_2 |
| B - NEEDLE VALVE | F - 6 OR 8 PORT GAS SAMPLING VALVE | J - CHARCOAL G.C. COLUMN |
| C - REACTOR WITH COARSE FRIT
AND SULFAMIC ACID SOLUTION | G - SPARGE GAS VENT | K - T.C. DETECTOR |
| D - SEPTUM INLET FOR PROPELLANT | H - LM_2 TRAP FOR CO_2
H₁ (COLLECT POSITION)
H₂ (BYPASS POSITION) | L - CARRIER GAS INLET |

sample for background CO₂ determination. Collect the sample for 45 min and then position the stopcocks so that the sparge gas bypasses the cooled portion of the trap. Position the gas sampling valve so that the carrier gas will flow through the bypass line of the trap. Warm the lower portion of the trap to ambient temperature. Inject the trapped gases into the gas chromatograph by rotation of the stopcocks to the correct position. Determine the CO₂ 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 0.25ml propellant sample into the septum inlet of the apparatus and allow 45 min for complete transfer of the CO₂ from the sulfamic acid bubbler to the trap. Repeat the trapped sample injection sequence described earlier for the background CO₂ determination and record the sample CO₂ peak height or area.

4.5.8.2 Propellant Transfer. To achieve accurate CO₂ results, all propellant transfers must be performed in a CO₂ - 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 a gas tight syringe for the analysis. The gas tight syringe shall be cleaned and dried between each propellant sample injection into the apparatus.

4.5.8.3 Calculation. Calculate the CO₂ level of the propellant as follows

$$\%CO_2 = (R_s - R_B) S$$

Where: $(R_s - R_B)$ = CO₂ response due to the sample minus CO₂ response due to the background, peak height or area
 S = Sensitivity, percent CO₂ per unit response

4.5.8. Calibration. Accurately weigh 0.130g of (NH₄)₂CO₃·H₂O into a tared glass vessel which will accept a vapor tight cap and will contain 100ml of liquid. Place the vessel in the transfer apparatus. Transfer 100ml of the propellant sample to the vessel, cap the vessel. Dissolve and thoroughly mix the ammonium carbonate solution. Remove the vessel from the transfer apparatus and weigh the container. Calculate the CO₂ added as follows:

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

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 CO₂ in the calibration standard. Calculate the CO₂ sensitivity of the apparatus as follows:

$$S = \frac{\%CO_2(\text{added})}{R_{std} - R_s}$$

where: $R_{std} - R_s$ = the response due

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to the CO_2 in the standard
minus the response due to the
sample, peak height or area.

4.5.8.5 Reagents and Equipment.

4.5.8.5.1 Reagents.

1. Helium, gaseous, conforming to Mil-P-27407, two cylinders.
2. Sulfamic Acid, aqueous, 90% saturated solution.
3. Bromocresol Green, indicator, several drops added to the sulfamic acid solution will provide a visual indication of when the solution is spent
4. Magnesium Perchlorate, anhydrous, granular.
5. Liquid Nitrogen.
6. Ammonium Carbonate Monohydrate, A.C.S. Reagent Grade.
7. Charcoal, coconut, 60/80 mesh, activated.
8. Water, distilled or deionized.

4.5.8.5.2 Equipment.

1. Reactor, sulfamic acid, constructed so that the volume is approximately 75cc., Borosilicate glass.
2. Drying tube, $\text{Mg}(\text{ClO}_4)_2$, approximately 6"x1/2", Borosilicate glass.
3. Trap, CO_2 , Borosilicate glass, constructed with 6mm O.D. tubing, the lower loop should contain glass beads to increase the condensing surface area.
4. Valve, gas sampling 6 or 8 port
5. Valve, needle, for regulation of the sparge gas.
6. Dewar, conical, to fit trap.
7. Gas chromatograph, equipped with a thermal conductivity detector, 6'x1/8" 60/80 mesh charcoal column.
8. Recorder potentiometric, 1mv span, 1 sec. F. S. pen speed.
9. Integrator, mechanical or digital, optional.

4.5.9 Other Volatile Carbonaceous Material. Other volatile carbonaceous material shall be determined by the following method

4.5.9.1 Procedure. Use the column, test conditions, and sample size

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described in 4.5.2.1. A Flame ionization detector shall be used instead of the thermal conductivity detector. 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 Calibration. Add 9.6 μ l of UDMH and 8.5 μ l of MMH to 75 ml 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.

$$\%UDMH = \frac{0.01 A_s}{A_c - A_s} \quad \text{Where.} \quad A_s = \text{Area of sample UDMH times its attenuation factor}$$

$A_c = \text{Area of calibration UDMH times its attenuation factor}$

$$\%MMH = \frac{0.01 A_s}{A_c - A_s} \quad \text{Where} \quad A_s = \text{Area of sample MMH times its attenuation factor}$$

$A_c = \text{Area of calibration MMH times its attenuation factor.}$

% 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
- c. Reagents = as required in 4.5.2

4.5.9.4.2 Equipment.

- a. Bottle = 4 oz., glass, narrow mouth, screw capped, plastic lined cap.
- b. Cylinder or syringe = 100 ml capacity graduated in 5 ml increments.
- c. Equipment = as required in 4.5.2, except gas chromatograph shall be equipped with a flame ionization detector.

4.6 Preparation for delivery inspection. The preservation, packaging, packing,

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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 170-190, or DOT special permit in accordance with 49 CFR 170.13 obtained by the shipper in conjunction with Commander, Headquarters Military Traffic Management and Terminal Service, Attn: Safety Division (TES) Washington DC 20315.

5.1.1 Approved drums. Drums which are approved by DOT for this propellant shall conform to DOT specifications 5, 5A, 5C, or 17 E (singletrip) of Types 304 or 347 stainless steel.

5.1.2 Approved cargo tanks. Cargo tanks which are approved by DOT for this propellant shall conform to DOT specifications MC 310, MC311, 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 111A100 W 6 of Types 304L or 347 stainless steel with molybdenum content not exceeding one-half of one percent. 111A100-W-6 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 Preparation of containers. 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 (6.2). 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 the service, at any required retest or overhaul, or an any time contamination is suspected.

5.2.2 Gaskets. Gaskets used to seal container openings shall be polytetrafluoroethylene or other material compatible with the propellant and approved for use by the procuring activity (6.2). The contractor shall assure that all gaskets are serviceable and furnish new gaskets when necessary so that a tight seal is assured.

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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 I, Grade A, at not less than atmospheric pressure.

5.4 Labeling and marking. 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 code shall be used.

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, NSN number, quantity, name of manufacturer, name of contractor (if different from manufacturer), date of manufacture, and lot identification number (6.2).

5.4.2 Precautionary label. A precautionary label prepared in accordance with MCA Manual L-1 shall be applied to each drum (6.4).

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-Water, 70/30". Any other name identification shall be obliterated by removing or overpainting.

6. NOTES

This section contains nonmandatory provisions only to assist both the contractor and buyer in the proper understanding and utilization of this specification.

6.1 Intended Use. The propellants covered by this specification are intended for use as fuel for gas generators.

6.2 Ordering Data. Purchasers should exercise any desired options offered herein, and procurement documents should specify the following.

6.2.1 Procurement Requirements:

- a. Title, number, type and date of this specification.
- b. Method of shipment, type and capacity of containers (5.1).
- c. Quantity by weight in pounds (avoirdupois).
- d. When other component limits are required (3.1).
- e. When test methods are other than specified (3.1).
- f. When particulate contamination requirements are to be waived (3.1).
- g. When inspection requirements are to be performed by other than the supplier (4.1).

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- h. When sampling is other than specified (4.2).
- i. When disposition of rejected product is required (4.4.3).
- j. When cleaning and repair schedule is required for leased or Government owned containers (5.2).
- k. When cleaning and repair of containers is to be other than as specified (5.2.1).
- l. When identification tag is to be other than specified (5.4.1).
- m. When approval of gasket material is required (5.2.2).
- n. When the color requirement is waived (3.4).

6.2.2 Contract Data Requirements. Data conforming to Data Item Description DI-T-3733, Quality Conformance Test Reports (Fuels), is a requirement for delivery in connection with this specification. The data item will be specified for delivery on the DD Form 1423.

6.3 Definition.

6.3.1 Particulate. The undissolved solids retained on the applicable filter membrane

6.4 Highway safety. To promote safety in the transportation of propellants in interstate commerce by motor vehicle, the shipper should assure that each driver possesses an MCA Chem-Card-Transportation Emergency Guide No. 66-71. A complete manual of cards or the individual cards are available from the Manufacturing Chemists' Association, 1825 Connecticut Avenue, N.W., Washington, DC 20009.

6.5 Precautionary labels. Precautionary labels are prepared in accordance with MCA Manual L-1. For those propellants which do not have specifically prescribed labels, the principles for the preparation of the labels are used. There does not have to be exact agreement between labels from different sources as long as the intent of the manual is complied with.

6.6 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 manufacture/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.

Custodians:	Preparing Activity
Air Force - 12	Air Force - 12
Review Activities:	
Air Force - 12, 19, and 68	Project No. 9135-F087

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