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MILITARY SPECIFICATION

PROPELLANTS, NITRIC ACID

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

1. SCOPE

1.1 Scope. This specification covers the requirements for nitric acid propellants.

1.2 Classification. The nitric acid shall be of the following types, as specified (6.2). For information on obsolete types see 6.1.1.

Type IIIA - Nominal 14 percent NO_2 content plus corrosion inhibitor.

Type IIIB - Nominal 14 percent NO_2 content, lower solids content, plus corrosion inhibitor.

Type IIILS - Limited storage propellant with nominal 14 percent NO_2 content, lower solids content, plus corrosion inhibitor, and limited iron content.

2. APPLICABLE DOCUMENTS

2.1 Defense Standardization Documents. 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.

STANDARDS

Military

MIL-STD-129 Marking for Shipment and Storage

MIL-STD-172 Color Code for Containers of Liquid Propellant

FSC 9135

(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.

Department of Transportation (DOT).

49 CFR 170	Administrative procedures of the Department of Transportation.
49 CFR 171-190, 46 CFR 146, & 14 CFR 103.	Hazardous Materials Regulations of the Department of Transportation. (For use of alternate commercial publications see 6.8).

Department of Defense (DOD).

DSAM 4145.3; Also AFM 71-4, TM 38-250, NAVAIR 15-03-500, or MCO 4145.3.	Packaging and Handling of Dangerous Materials for Transportation by Military Aircraft.

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402).

Manufacturing Chemists' Association (MCA).

Manual L-1	A Guide for the Preparation of Warning Labels for Hazardous Chemicals (1961).

(Application for copies should be addressed to the Manufacturing Chemists' Association, Inc., 1825 Connecticut Avenue, N.W., Washington, D.C. 20009).

American Society for Testing and Materials (ASTM).

D 1068-68	Standard Methods of Test for Iron in Industrial Water and Industrial Waste Water.
E 29-67	Recommended Practices for Designating Significant Places 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 composition and physical properties. The chemical composition and the physical properties of the propellant shall conform to the requirements specified in table I.

TABLE I. Chemical Composition & Physical Properties

Constituent	Type IIIA	Type IIIB	Type IIILS	Test Paragraph
HNO_3, percent by weight	81.6 - 84.8	81.6 - 84.8	83.6 - 86.3	4.5.9
NO_2, percent by weight	14 $\pm$ 1	14 $\pm$ 1	14 $\pm$ 1	4.5.3
HF, percent by weight	0.7 $\pm$ 0.1	0.7 $\pm$ 0.1	0.7 $\pm$ 0.1	4.5.4
H_2O, percent by weight	1.5 - 2.5	1.5 - 2.5	0.5 max.	4.5.8
Fe_2O_3, percent by weight, maximum.	not specified	not specified	0.0015	4.5.7
Solids, percent by weight, maximum as nitrates	0.10	0.04	0.04	4.5.6
Specific Gravity, $60^oF/60^oF$ Minimum	1.564	1.564	1.572	4.5.5
Specific Gravity, $60^oF/60^oF$ Maximum	1.575	1.575	1.582	4.5.5

3.2 Limiting values. The following applies to all specified limits in this specification. For purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand 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 Qualitative. The propellant shall be a single phase liquid (6.4) when examined visually by transmitted light, and the IRFNA spectrum resulting from the water analysis (4.5.8 & figure 2) shall contain the 1440 mμ peak which identifies the presence of nitric acid.

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 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 tests 4.4.1

(b) Sampling tests 4.4.2

4.4.1 Individual tests. The propellant shall be subjected to the following test as specified in 4.5.

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 following tests as specified in 4.5.

(a) Nitrogen dioxide 4.5.3

(b) Hydrogen fluoride. 4.5.4

(c) Specific gravity 4.5.5

(d) Total solids 4.5.6

(e) Iron content 4.5.7

(f) Water. 4.5.8

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 is used to fill shipping containers directly from the process output. A continuous process shall be the production of propellant by 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 150 milliliters (ml) of propellant. 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.

4.4.2.1.3 Drums. The number of drums selected for sampling from each lot shall be in accordance with table II. The first and last containers to be filled within a given lot shall be sampled. Other containers may be selected at random. If more than one lot is represented in the shipment, then each lot represented shall be treated as a separate shipment for sampling purposes. The contents of each selected drum for sampling shall be thoroughly mixed by rolling and inverting immediately prior to sampling. The samples may be obtained in any convenient manner. Each sample shall be subjected to all the tests of this specification.

TABLE II. Sampling for Tests

Number of containers in lot.	Number of containers to be sampled.
2-25	2
26-150	3
151-1200	5

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 from a point 6 to 12 inches of the bottom of the shipping container and shall be tested to determine compliance with this specification.

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. 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. Disposition or retest of rejected product shall be as specified by the procuring activity (6.2).

4.5 Test methods.

4.5.1 Examination of product. The propellant shall be visually examined to determine compliance with the requirement specified herein. Examination to insure that the propellant conforms to paragraph 3.3 shall be conducted after 25 ml of sample has been transferred to a 100-ml polychlorotrifluoroethylene or equal vessel.

4.5.2 Neutralization. The acid sample shall be neutralized by the following procedure.

4.5.2.1 Sample preparations. Transfer 1 ml of acid into a tared 2-ml polyethylene or polypropylene weighing vial and close the top. Weigh the vial with the sample and determine the sample weight. Sample sizes shall average about 1.3 g when using 0.5 N alkali.

4.5.2.2 Procedure. The weighed ampoule shall be placed into a heavy-walled, glass stoppered 500-ml iodine flask, containing a required measured amount of 0.5 N sodium hydroxide which is in excess of that required for neutralization. The amount of alkali needed shall be calculated after the sample weight is known. Cool the contents of the iodine flask in a salt-water-ice bath for 5 minutes. Displace the air in the flask with gaseous nitrogen, conforming to MIL-P-27401. Immerse the plastic vial in a low temperature bath and freeze the liquid sample. Open the top of the vial, drop it into the flask after the air above the solution has been displaced, and quickly insert the glass stopper. The stopper shall be held in place with the thumb, and the flask shaken vigorously until the ampoule is opened. The flask shall be allowed to stand for 15 minutes at room temperature with occasional shaking during this time. A small quantity of distilled water shall be poured into the lip of the flask, the stopper loosened to allow the water to run into the flask and the stopper shall be rinsed with a stream of distilled water, also into the flask. Add 3 to 5 drops of phenolphthalein indicator to insure that the contents of the flask are slightly alkaline with respect to phenolphthalein.

4.5.2.3 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.2.

(a) Reagents

(1) Sodium hydroxide solution, 0.5 N:
Dissolve 20 g of ACS reagent grade, low carbonate, NaOH in distilled water in a 1000-ml volumetric flask. Allow

to cool and dilute to the mark with distilled water. Store in polyethylene and insure the exclusion of atmospheric carbon dioxide.

(2) Distilled water.

(3) Phenolphthalein indicator.

(4) Gaseous nitrogen, conforming to MIL-P-27401.

(b) Equipment

(1) Flask, iodine, heavy walled, glass stoppered, 500-ml capacity.

(2) Ampoules, polyethylene or polypropylene, as required.

(3) Flasks, volumetric, 1000-ml capacity.

(4) Ice bath, salt-water.

(5) Low temperature bath, difluorodichloromethane.

4.5.3 Nitrogen dioxide. The nitrogen dioxide content of all types of propellant shall be determined by the following procedure:

4.5.3.1 Procedure. The slightly alkaline sample of 4.5.2 shall be transferred quantitatively to a 250-ml volumetric flask and diluted to volume with distilled water. A 50-ml aliquot of this solution shall be withdrawn and transferred to a 250-ml flask and placed in the salt-water ice bath. The remaining solution shall be transferred to a plastic screw-cap container and reserved for tests in 4.5.4. One ml of 1 N H_2SO_4 shall be added with stirring. Add 50 ml of 0.1 N ceric solution without stirring and if the solution does not turn yellow, an additional 5 ml shall be added. The beaker shall be placed on the stirring platform and the electrode assembly immersed, and a blanket of gaseous nitrogen conforming to MIL-P-27401, Type I, shall cover the contents. Start the stirring motor and titrate the excess ceric solution with standardized 0.05 N ferrous ammonium sulfate potentiometrically using the millivolt scale of a pH meter or a recorder. A plot of volume (ml) versus millivolts (mv) shall be drawn from which the end point can be determined.

4.5.3.2 Calculation. The NO_2 content shall be calculated by the following formula:

$$\text{Weight \% } NO_2 = \frac{[(\text{ml } Ce^{4+} \times N) - (\text{ml } Fe^{2+} \times N_1)] \times 4.601}{W \times 0.2}$$

Where:

N = normality of the ceric solution

N_1 = normality of ferrous ammonium sulfate

W = original weight in grams (4.5.2.1)

4.5.3.3 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.3.

(a) Reagents

(1) Standard ceric solution 0.1 N: Dissolve exactly 54.878 g of dry Primary Standard-grade ceric ammonium hexanitrate $[(NH_4)_2\ Ce(NO_3)_6]$ in a 1000-ml volumetric flask with distilled water and dilute to the mark. The ceric nitrate is dried for 2 hours at 140°F (60°C) in a vacuum oven. Store the solution in a polyethylene bottle with a gaseous nitrogen atmosphere. Solution which has been prepared in excess of 7 days shall be standardized weekly before use with NBS primary standard grade arsenic trioxide using ferroin as the indicator.

(2) Standard ferrous solution, 0.05 N: Dissolve 19.608 g of ferrous ammonium sulfate hexahydrate, $(Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O)$, with 0.5 N sulfuric acid in a 1000-ml volumetric flask, and dilute to the mark with the same sulfuric acid solution. Standardize the ferrous sulfate reagent against 25 ml of the 0.1 N ceric solution in triplicate, using the potentiometric procedure of 4.5.3.1.

(3) Sulfuric acid solution, 0.5 N and 1 N: Dilute 14 ml and 28 ml respectively of concentrated ACS reagent-grade H_2SO_4 to 1000 ml with distilled water.

(4) Nitrogen, gaseous, conforming to MIL-P-27401, Type I.

(b) Equipment

(1) Volumetric flasks, 250-, 1000-, and 2000-ml.

(2) Volumetric pipets, 50- and 25-ml.

(3) Buret, 50-ml, 0.1-ml graduations, 2 each.

(4) Ice bath, salt-water.

(5) Flasks, 250- to 400-ml.

(6) Stirring motor and magnetic polytetrafluoroethylene covered stirring bar.

(7) Plastic bottle, 1 pt, polyethylene, or equivalent.

(8) Electrode assembly, platinum and saturated calomel.

(9) pH meter, equipped with a millivolt scale.

(10) Recorder, optional, Varian G-11, or equivalent.

4.5.4 Hydrogen fluoride. The hydrogen fluoride content of all types of propellant shall be determined by the following procedure.

4.5.4.1 Procedure. A 100-ml aliquot of the slightly alkaline solution of 4.5.3.1 shall be transferred to a 500-ml volumetric flask, diluted to the mark with distilled water, and mixed. A specific selected volume from 25 ml to 50 ml is transferred immediately to a plastic 100- to 200-ml beaker. The pH is adjusted to 5.5 by addition of an equal volume of citrate buffer. Check the calibration curve of the specific ion meter using the 2 ppm and 4 ppm fluoride standard solution. As soon as stable and repetitive readings are obtained, the meter system is ready for measuring the sample. Immerse the fluoride specific ion and the silver/silver chloride reference electrodes in the sample solution. Turn on the stirring motor and allow at least 15 seconds before taking a reading. Repeat the calibration check procedure using the 2 ppm and 4 ppm fluoride standard solutions. Obtain the ppm F^- ($\mu g\ F^-/ml$) from the calibration curve.

4.5.4.2 Calculation. The hydrogen fluoride content shall be calculated by the following formula:

$$\text{Hydrogen fluoride, wt \%} = \frac{0.264\ a}{W}$$

Where:

a = $\mu g\ F^-$/ml from calibration curve

W = original weight in grams (4.5.2.1)

4.5.4.3 Calibration curve. A calibration curve shall be prepared as follows. Place a 100-ml calibration line on four clean 4 oz plastic screw-cap bottles, using 100 ml of distilled water. Discard over half of the water and pipet, or measure from a microburet 1.0, 2.0, 4.0, and 8.0 ml of the working NaF standard, (100 $\mu g\ F^-$/ml), into the four marked bottles. Dilute to the mark with citrate buffer and mix. Using the same electrode and meter specified in 4.5.4.1, obtain a stable reading from each bottle and plot meter reading versus micrograms of fluoride per milliliter (microgram F^-/ml) using distilled water as zero concentration.

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

(a) Reagents

(1) Citrate buffer: Combine in a beaker 57 ml glacial acetic acid, 58 g NaCl, and 0.3 g sodium citrate with 500 ml

of distilled water. Adjust the pH to between 5.0 and 5.5 with NaOH (pellets). Transfer the mixture to a 1000-ml volumetric flask and dilute to the mark with distilled water.

(2) Stock standard F^- solution: Dry several grams of ACS reagent grade sodium fluoride overnight in a vacuum oven at 212°F (100°C). Weigh out exactly 2.2105 g of dried NaF and dissolve it in distilled water contained an a 1000-ml volumetric flask and dilute to the mark. Transfer the solution to a plastic screw-cap bottle. This solution contains 1000 ppm F^- (1.0 mg F^-/ml).

(3) Working standard F^- solution: Pipet 10.0 ml of solution (2) into a 100-ml volumetric flask and dilute to the mark with distilled water. Transfer to a plastic screw-cap bottle for storage. This solution contains 100 ppm F^- (100 µg F^-/ml).

(b) Equipment

(1) Pipets, 1-, 2-, 4-, 8-, 10-, 25-, 50-, and 100-ml.

(2) Microburet, 10-ml, 0.01 graduations.

(3) Plastic bottles, 4 oz and 1 qt.

(4) Plastic beakers, 100- and 200-ml.

(5) Electrodes, silver/silver chloride and F^- specific ion.

(6) pH meter with glass and SCE electrodes.

(7) Specific ion meter, Orion Model 401 or 404, or equivalent.

(8) Volumetric flasks, 100-, 500-, and 1000-ml.

4.5.5 Specific gravity. The specific gravity shall be determined at 15.6°C/15.6°C (60°F/60°F) by any convenient standard method, such as a hydrometer and the specific gravity shall conform to the limits in table I. Because of HF attack, periodic calibration will be required.

4.5.5.1 Equipment. The following equipment shall apply as a test condition of 4.5.5.

(1) Cylinder, graduated, 100-ml.

(2) Hydrometer, 1.49-1.60 NBS calibrated.

(3) Thermometer, °F or °C, .2°F or .1°C increments, NBS calibrated.

4.5.6 Total solids. The total solids of all types of propellant shall be determined by the following procedure.

4.5.6.1 Procedure. The sample shall be thoroughly shaken. Transfer exactly 25.0 ml of propellant to a clean platinum crucible accurately tared to the nearest 0.1 mg. Add 1 ml of concentrated sulfuric acid and slowly evaporate the acid on a hotplate in a fume hood until dense fumes of sulfur trioxide evolve. Transfer the crucible from the hotplate to a triangle over a Meeker burner to continue the decomposition of the sulfates. Ignition may be carried out at 1652°F (900°C) for 30 minutes to constant weight in a muffle furnace or over a Meeker burner. Save the residue for the iron analysis in 4.5.7.

4.5.6.2 Calculation. The residue shall be calculated by the following formula:

$$\text{Residue \% by weight} = \frac{g \times 100 \times a}{25 \times \text{Sp. G.}}$$

Where:

a = 2.09 for aluminum containers or
1.52 for steel containers.

4.5.6.3 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.6.

(a) Reagents

Sulfuric acid: ACS reagent grade, 98% H_2SO_4.

(b) Equipment

(1) Platinum crucible, 50- to 100-ml.

(2) Hotplate, electrical.

(3) Triangle, silica.

(4) Tripod.

(5) Meeker burner, or equivalent.

(6) Analytical balance, 0.1 mg sensitivity, 200 g capacity.

4.5.7 Iron content. The iron content of type IIILS propellant shall be determined by the following method or ASTM D-1068, Method A, paragraph 12. In case of dispute the following method shall be used.

4.5.7.1 Procedure. Five ml of 6 N HCl shall be added to the dry residue of 4.5.6 to dissolve it. It shall then be evaporated to near dryness on a 250°F (120°C) hotplate. The contents of the platinum evaporating dish shall be quantitatively transferred to a 25-ml volumetric flask with several rinsings of 0.1 N HCl solution. The sample shall be diluted to the 25-cc mark with 0.1 N HCl and mixed by inverting several times. Determine the percent absorption of the sample at an Fe wavelength closest to 2480Å on a suitable atomic absorption spectrophotometer, after setting the 0 percent absorption with the 0.1 N HCl solution. Convert the percent absorption reading to absorbance (A) and find the ppm Fe (µg/ml) for the sample from the calibration curve previously constructed.

4.5.7.2 Calculation. The iron oxide (Fe_2O_3) content of the sample shall be calculated as follows:

$$\text{percent } Fe_2O_3 = \frac{\text{ppm Fe (from calibration curve) x 1.43}}{\text{specific gravity x } 10^4}$$

4.5.7.3 Calibration. A calibration curve shall be constructed by the following procedure. Prepare 5 ppm Fe, 10 ppm Fe, and 20 ppm Fe standards by taking 0.5 ml, 1.0 ml, and 2.0 ml of the 1000 ppm Fe Stock Standard solution respectively, placing the aliquots into each of three 100-ml volumetric flasks and diluting to the mark with the 0.1 N HCl solution. Transfer the dilute standards to plastic screw-cap bottles. Determine the percent absorption of the three Fe standards at 2480Å, which is the same Fe line as for the sample, using the 0.1 N HCl solution to set the instrument at 0 percent absorption. Convert the percent absorption to absorbance (A) and plot A versus ppm Fe on linear graph paper. Draw a smooth curve through the origin. Replace the dilute standards whenever they deviate from the points on the freshly prepared calibration curve.

4.5.7.4 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.7.

(a) Reagents

(1) Hydrochloric acid, 6 N: Add 50 ml distilled/deionized water to 50 ml ACS reagent grade 37% (average) concentrated HCl. Store in a plastic bottle.

(2) Hydrochloric acid, 0.1 N: Dilute 2 ml of 6 N HCl to the mark with distilled/deionized water in a 1000-ml volumetric flask. Store in a plastic bottle.

(3) Iron standard solution, 1000 ppm: Weight 1.000 g Fe wire or shot accurately. Transfer to a 1000-ml volumetric flask and add 17 ml 6 N HCl solution. Heat on hotplate until the Fe is dissolved. Dilute to the mark with distilled/deionized water.

(4) Distilled/deionized water.

(b) Equipment

(1) Volumetric flasks, 25-, 100-, and 1000-ml, as required.

(2) Hotplate, controllable heat settings.

(3) Atomic absorption spectrophotometer, Perkin Elmer Model 290 or 303, or equivalent, with an Fe hollow cathode lamp.

(4) Plastic bottles, screw-cap, 4 oz, as required.

4.5.8 Water. The water content shall be determined by the following procedure.

4.5.8.1 Procedure. Obtain the absorption spectrum of the sample between 1500 and 1350 mμ versus CCl_4 both contained in 5-mm cells constructed in accordance with figure 1. Determine the absorbance (ΔA) at 1405 mμ in accordance with the technique illustrated by figure 2. The water content is determined from the calibration curve prepared in accordance with 4.5.8.3 and the following calculation.

4.5.8.2 Calculation. Calculate the weight percent water in the sample as follows:

$$\text{Percent water} = \frac{D}{d} \times 100$$

Where:

D = g H_2O/ml CH_3CN from calibration curve

d = specific gravity of sample (4.5.5)

4.5.8.3 Calibration. Calibrate the spectrophotometer using the same cells used for the sample analysis. Prepare a series of water standards containing 0.00, 0.01, 0.02, 0.03, 0.04, 0.06 g H_2O/ml of acetonitrile (CH_3CN) solution and a series of reference beam compensating solutions substituting tetrachloromethane (CCl_4) for the water. Prepare the standards in accordance with the following table:

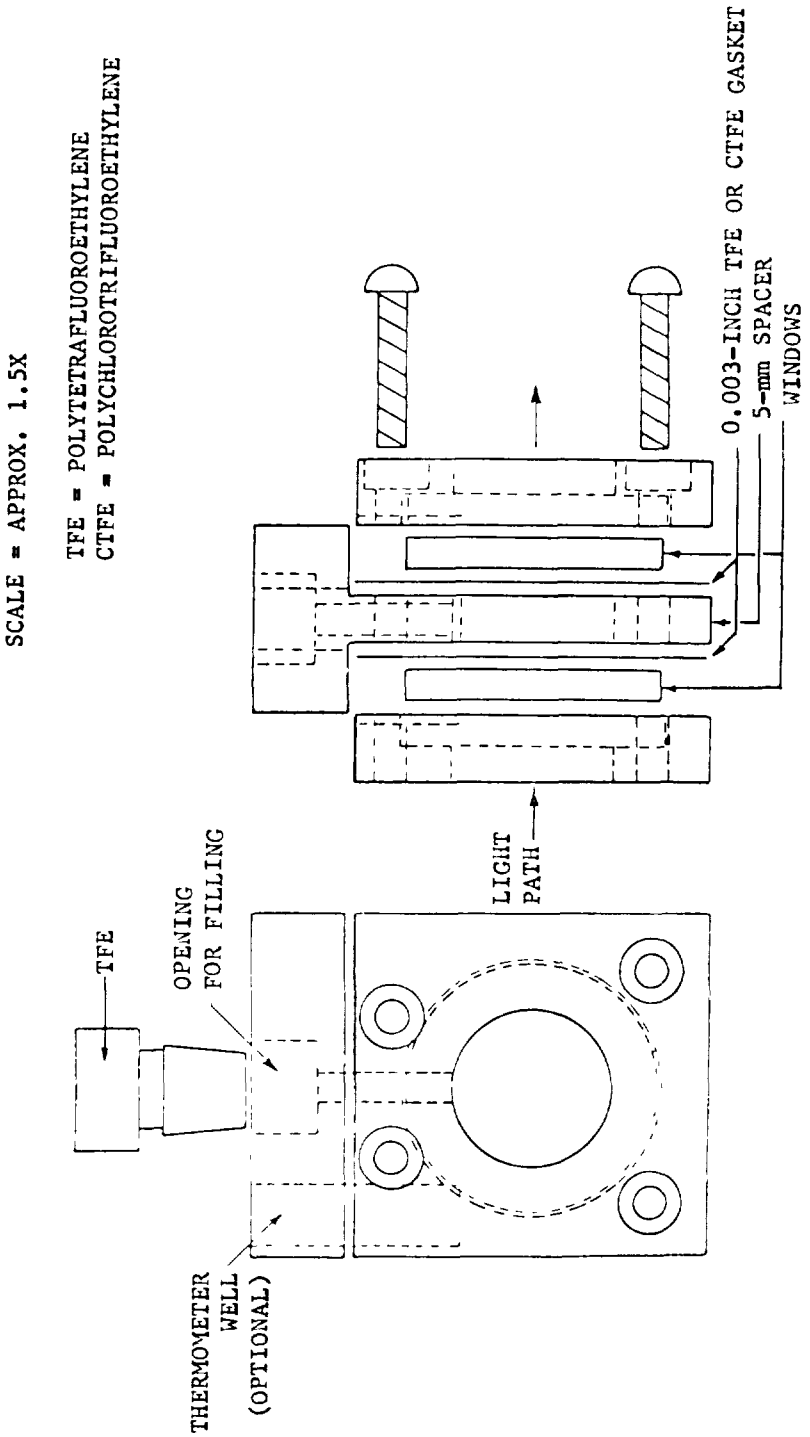

Figure 1. Stainless Steel Absorption Cell with Sapphire Windows.

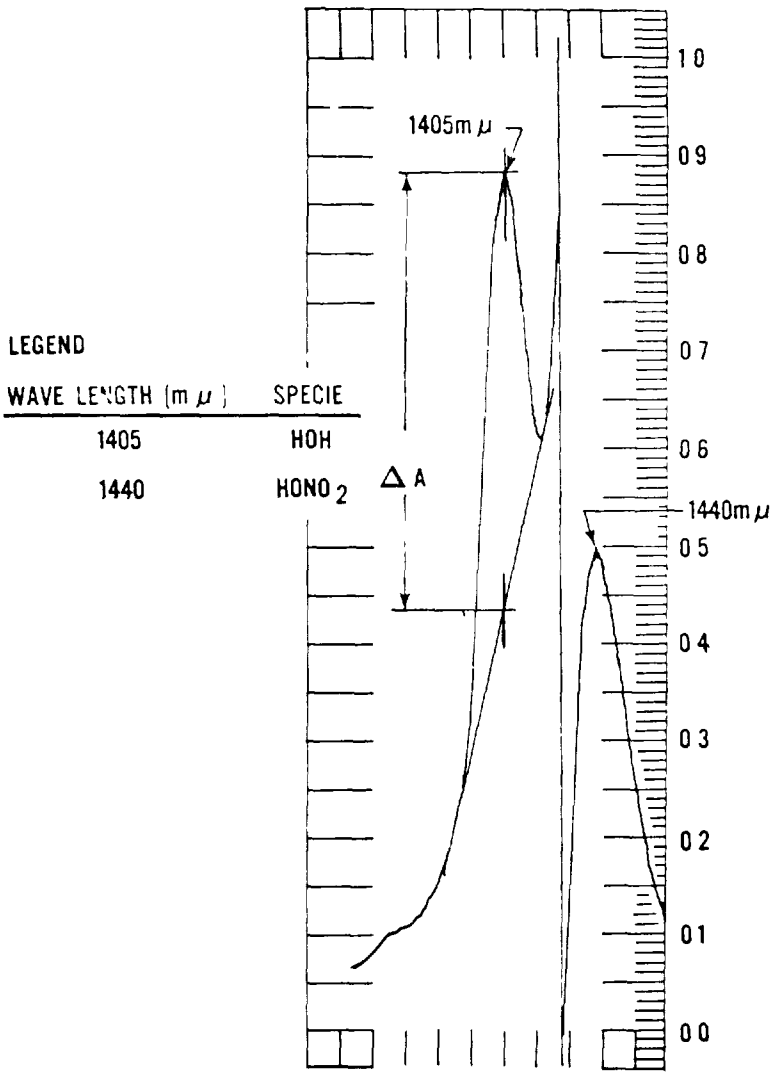

Figure 2. A Typical IRFNA Spectrum Versus CCl_4

TABLE III. Standards

g H_2O/ml CH_3CN solution	g H_2O or ml CCl_4	ml CH_3CN solution
0.00	0.00	50
0.01	0.50	49.5
0.02	1.00	49.0
0.03	1.50	48.5
0.04	2.00	48.0
0.06	3.00	47.0

Determine the absorbance of each H_2O/CH_3CN solution versus the matching CCl_4/CH_3CN solution between 1500 mμ and 1350 mμ in the 5-mm cells. Plot a curve through the points showing the absorbance (ΔA) versus g H_2O/ml CH_3CN.

4.5.8.4 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.8.

(a) Reagents

(1) Water, distilled.

(2) Acetonitrile, ACS reagent grade

(3) Carbon tetrachloride, ACS reagent grade

(b) Equipment

(1) Spectrophotometer, double beam, recording, range from 2500 mμ to 350 mμ or better.

(2) Cell, absorption, 2 each (figure 1).

(3) Syringe, polytetrafluoroethylene, 2.5 ml.

(4) Flask, 50-ml, volumetric.

(5) Balance, 500 g capacity, 0.01 g sensitivity.

4.5.9 Final calculations. The final calculations shall be performed by the following procedure.

4.5.9.1 Procedure.

$$\text{Percent } HNO_3 = 100 - [\%HF + \% \text{ residue} + \% NO_2 + \%H_2O]$$

4.6 Preparation for delivery inspection. The preservation, packaging, packing, and marking for shipment and storage of the propellant shall be inspected to determine compliance with the requirements of

section 5 of this specification.

5. PREPARATION FOR DELIVERY

5.1 Packaging. The product furnished under this specification is a hazardous material as defined and regulated by the Department of Transportation (DOT) regulations. All packaging to be shipped commercially by any mode of transportation shall comply with the requirements of DOT regulations 49 CFR 171-190, or DOT special permit obtained in accordance with 49 CFR 170.13 by the shipper in conjunction with the Commander, Headquarters Military Traffic Management and Terminal Service, Attn: Safety Division (TES), Washington, D.C. 20315. All packaging to be shipped by military air shall comply with DSAM 4145.3 (AFM 71-4). The product shall be packaged in containers and unit quantities as specified by the procuring activity (6.2 and 6.3).

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 expense. Leased or Government owned containers which the contractor finds unsuitable for filling shall be reported and subsequently cleaned and repaired in accordance with the instructions and schedule established in the contract or purchase order (6.2).

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, at any required retest or overhaul, or at any time contamination has been found.

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. 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.

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

5.4.2 Precautionary label. A precautionary label prepared in accordance with MCA Manual L-1 shall be applied to each drum (6.6).

5.4.3 Container color code. Unless otherwise specified by the procuring activity, each drum shall be color coded in accordance with MIL-STD-172 and the exact name identification to be marked on the outside of the drum shall be "Nitric Acid". 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 oxidizers in rocket engines. Type IIILS is intended for special purposes and will not be used for applications where storage life is a criterion because of its tendency to change composition.

6.1.1 Obsolete classifications. Propellant types I, IA, and III contained in MIL-P-7254E have become obsolete. Propellant types II and IV have previously become obsolete.

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 and 6.3).

(c) Quantity by weight in pounds (avoirdupois).

(d) When inspection requirements are to be performed by other than the supplier (4.1).

(e) When sampling is other than specified (4.4.2.1.2, 4.4.2.1.4, and 4.4.2.1.5).

(f) When disposition or retest of product is required (4.4.3).

(g) When cleaning and repair schedule is required for leased or Government owned containers (5.2).

(h) When cleaning and repair of containers is to be other than as specified (5.2.1).

(i) When approval of gasket material is required (5.2.2).

(j) When identification tag is to be other than as specified (5.4.1).

(k) When the container color code is to be waived (5.4.3).

6.2.2 Contract data requirements. Data conforming to Data Item Description DI-T-138-1, 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. In addition to the copies required by the procuring activity, one copy of each report should be addressed to AFRPL (RPORS), Edwards, CA 93523.

6.3 Containers. As of the date of this specification, the following listed containers are considered acceptable for military use and are approved for nitric acid by DOT regulations as specified in 49 CFR 173.268 or DOT special permits as stated. Types IIIA and IIIB propellant should be, on a preferential basis and to the greatest extent feasible, shipped in aluminum containers; however, stainless steel (300 series) may be used. Type IIILS propellant should be shipped exclusively in aluminum containers.

(a) Sample quantities as specified in DOT special permit no. 2100.

(b) Aluminum drums of specification DOT 42B and conforming to MIL-D-4303.

(c) Tank cars of specifications DOT 103A-AL-W, 103C-W, or 103C-AL.

(d) Cargo tanks of specifications DOT MC 310, MC 311, or MC 312.

6.4 Definition.

a. Single phase liquid. A single phase liquid is devoid of any visible foreign liquid, but may contain solid material as permitted within this specification.

6.5 Highway safety. To promote safety in the transportation of propellants in interstate commerce by motor vehicle, each product contractor or shipper should assure (and provide if necessary) that each driver possesses an MCA Chem Card - Transportation Emergency Guide No. CC-3. A complete manual of cards or the individual cards are available

from the Manufacturing Chemists' Association, 1825 Connecticut Avenue, N.W., Washington, D.C. 20009.

6.6 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.

6.7 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 adaquate control and disposal methods for situations which are likely to develop in any of the phases.

6.8 Alternate publications. The following commercial publications may be used in lieu of or in conjunction with 49 CFR 171-190, 46 CFR 146, and 14 CFR 103 as appropriate as the content is identical or in the case of airlines is supplimentary.

(a) Agent T. C. George's Tariff No. 23, Hazardous Materials Regulations of the Department of Transportation. Available from Association of American Railroads, Bureau of Explosives, 63 Vesey Street, New York, N.Y. 10007.

(b) Agent Fruend's Tariff, Hazardous Materials Regulations of the Department of Transportation. Available from American Trucking Associations, Inc., 1616 P Street, N. W. Washington, D.C. 20036.

(c) Tariff 6D, Transportation of Restricted Articles by Air. Available from Airline Tariff Publishers, Inc., 1825 K Street, N.W. Washington, D.C. 20006.

Custodians:

Army - MI
Navy - AS
Air Force - 12

Review Activities:

Army - MI
Navy - AS
Air Force - 19 and 68
NASA

Preparing Activity:

Air Force - 12

Civilian Agency Interest:

NAS

Project No. 9135-0022

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 119-R004

INSTRUCTIONS

This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity *(as indicated on reverse hereof)*.

SPECIFICATION

MIL-P-7254F, PROPELLANTS, NITRIC ACID

ORGANIZATION *(Of submitter)*	CITY AND STATE

CONTRACT NO	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT
		$

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ SUBCONTRACT

1 HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?

A GIVE PARAGRAPH NUMBER AND WORDING.

B RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES.

2 COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID

3. IS THE SPECIFICATION RESTRICTIVE?

☐ YES ☐ NO IF "YES", IN WHAT WAY?

4 REMARKS *(Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)*

SUBMITTED BY *(Printed or typed name and activity)*	DATE

DD, FORM 1 APR 63 1426 REPLACES NAVSHIPS FORM 4863, WHICH IS OBSOLETE C-8279

FOLD

AFRPL (RPORS)
EDWARDS, CALIFORNIA 93523

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Director
Air Force Rocket Propulsion Laboratory (RPORS)
Edwards, California 93523

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