INCH-POUND

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SUPERSEDING
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NOTICE 2
14 October 2005

PERFORMANCE SPECIFICATION

PROPELLANT, NITROGEN TRIFLUORIDE

Reactivated after *(03Jan2011)* and may be used for new and existing design acquisitions

Comments, suggestions, or questions on this document should be addressed to HQ Air Force Petroleum Agency (AFPET)/PTPT, 2430 C Street, Bldg 70, Area B, Wright-Patterson AFB OH 45433-7632 or e-mailed to AFPET.AFTT@wpafb.af.mil. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at https://assist.daps.dla.mil.

AMSC N/A FSC 9135

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CONTENTS

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 nitrogen trifluoride (NF_3) propellant.

1.2 Classification. Nitrogen trifluoride will be of the following type as specified (see 6.2):

Type I - Gaseous

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3, 4, or 5 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3, 4, or 5 of this specification, whether or not they are listed.

2.2 Government documents.

2·2.1 Specifications, standards, and handbooks. The following specifications, standards and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-PRF-27407	-	Propellant Pressurizing Agent, Helium

DEPARTMENT OF DEFENSE STANDARDS

MIL-STD-1411	-	Inspection and Maintenance of Compressed Gas Cylinders

(Copies of these documents are available online at https://assist.daps.dla.mil/quicksearch/ or from the Standardization Document Order Desk, 700 Robbins Avenue, Bldg 4D, Philadelphia PA 19111-5094.)

2.3 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

ASTM INTERNATIONAL

ASTM E29	-	Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
ASTM F307	-	Standard Practice for Sampling Pressurized Gas Analysis

(Copies of these documents are available online at http://www.astm.org or the ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken PA 19428-2959)

KENNEDY SPACE CENTER

KSC-SPEC-P-0022 - Solvent, Cleaning, 1,3-Dichloro-1,1,2,2,3-Pentafluoropropane, HCFC-225G, Specification For

(Copies of this document is available online at http://standards.nasa.gov/ or from NASA Technical Standards Program Office, Marshall Space Flight Center, Marshall Space Flight Center, AL 35812).

ASME INTERNATIONAL

ASME B40.100-2005 - Pressure Gauges and Gauge Attachments

(Copies of this document is available online at http://www.asme.org/ or may be obtained from ASME Information Central Order/Inquiries PO Box 2300, Fairfield, NJ 07007-2300, (US/Canada (800)843-2763; outside North America (973)882-1170).

2.4 Order of precedence. Unless otherwise cited herein or in the contract, in the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Chemical composition and physical properties. The chemical composition and physical properties of the propellant shall conform to Table I when tested in accordance with (IAW) the test methods in 4.5.

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 digit used in expressing the specification limit according to the rounding-off method of ASTM Practice E29, Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications.

3.3 Filled containers.

3.3.1 Cylinders. Gaseous NF_3 cylinders shall be prepared in accordance with procedures for oxygen in MIL-STD-1411 with the following exceptions:

a. The odor test is omitted.

b. The cylinder and tube valve outlets shall be according to Compressed Gas Association Valve Outlet 330. The main manifold valve on the tube bank trailers shall be Circle Seal Valve P/N MV 3308P/MV 3308P-P unless otherwise specified

3.3.2 Filling pressure. The container filling pressure shall not differ from that required by the contract by more than 1.0% at 70°F when tested as specified in 4.2.1.1. In no case shall the filling pressure exceed the rated service pressure of the container.

3.3.3 Leakage. Containers should not leak when tested IAW 4.2.1.2.

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

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

Composition	Limit	Test Paragraph
Purity, Nitrogen trifluoride (NF_3), % Vol (min)	99.0	4.5.1
Tetrafluoromethane (CF_4), % Vol (max)	0.5	4.5.2
Carbon Dioxide (CO_2), % Vol (max)	0.1.	4.5.2
Nitrous Oxide (N_2O), % Vol (max)	0.1	4.5.2
Sulfur Hexafluoride (SF_6), % Vol (max)	0.1	4.5.2
Oxygen (O_2) + Argon (Ar), % Vol (max)	0.1	4.5.3
Nitrogen (N_2), % Vol (max)	0.1	4.5.3
Carbon Monoxide (CO), % Vol (max)	0.1	4.5.3
Total Reactive Fluoride (as HF), % Vol (max)	0.1	4.5.4
Water (H_2O), ppmv (max)	5	4.5.5

4. VERIFICATION

4.1 Classification of inspections. The inspections shall be classified as conformance inspections.

4.2 Conformance inspection. Quality conformance tests shall consist of the following:

a. Individual tests ———————————— 4.2.1

b. Sampling tests ———————————— 4.2.2

4.2.1 Individual tests. Each container shall be subjected to the following tests:

4.2.1.1 Filling pressure. The container shall be tested for proper filling pressure by using an ASME B40.1 type pressure gauge with a minimum of a 2A accuracy grade; calibration must be NIST traceable.

4.2.1.2 Leakage. Each container shall be tested for leaks at the neck threads, stem packing, and pressure relief device of the valve with leak detection fluid. Valve seat leakage shall be tested by means of a tube from the valve outlet to a container of liquid.

4.2.2 Sampling for tests. When more than one container (cylinders or tubes) is filled from the same lot, the number of containers selected for sampling from that lot shall be IAW Table II. The first and last containers to be filled within a given lot shall be sampled. Other samples may be selected at random. The propellant from each container sampled shall constitute a separate sample.

TABLE II. Sampling for tests.

Number of containers in lot	Number of containers to be sampled
1	1
2 - 40	2
41 - 70	3
70 and over	4

4.3 Sampling plan.

4.3.1 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 product 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 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 by runs 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 homogenous 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.

4.3.1.1 Sample size. Each sample shall be of sufficient size to conduct all the quality conformance tests as specified herein.

4.3.1.2 Sampling method. Unless otherwise specified, the propellant shall be sampled IAW ASTM F307. All apparatus used shall be made of suitable materials. Each sample obtained shall be representative of the propellant as a whole.

4.4 Rejection. When any sample of the propellant tested in accordance with 4.5 fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected.

4.5 Test methods. The contractor's own test methods may be used if approved by the procuring activity. In case of a dispute, the following shall be the referee methods.

4.5.1 Purity. Calculate the nitrogen trifluoride (NF_3) purity as follows:

$$NF_3\ \%Vol = 100 - \Sigma\%i$$

where,

$\Sigma\%i$ = The sum of the impurities found in 4.5.2 through 4.5.5.

4.5.2 Tetrafluoromethane, carbon dioxide, nitrous oxide, and sulfur hexafluoride. Tetrafluoromethane (CF_4), carbon dioxide (CO_2), nitrous oxide (N_2O) and sulfur hexafluoride (SF_6) in the propellant shall be determined by gas chromatography.

4.5.2.1 Instrument Parameters. The analyst may vary the column dimensions, carrier gas flow rate, and temperatures to optimize the analysis.

Detector:

Thermal conductivity detector, discharge ionization detector, or a pulsed discharged helium ionization detector.

Column:

Packing:	Super Q, 80/100 (or equivalent)
Length:	4.9 m (16 ft)
ID:	1.75 mm (1/16 in.)
OD:	3.2 mm (1/8 in.)
Column material:	stainless steel

Carrier Gas / Flow Rate:

Helium (MIL-PRF-27407) / 19 mL/min

Temperatures:

Detector:	100°C
Oven:	42°C
Gas sampling valve:	42°C

4.5.2.2 Calibration. Prepare a gaseous standard of helium containing at least 0.1% NF_3 and the components being analyzed in a concentration between one tenth and 10 times the specification limit or of the predicted measurement in the propellant sample. Analysis of the calibration standard must show a clear separation of each specified component from all other components and from NF_3.

4.5.2.3 Procedure. Inject the calibration standard into the column using a gas-sampling valve. Record the retention times and peak areas for each component. The order of elution is tetrafluoromethane, nitrogen trifluoride, carbon dioxide, nitrous oxide, and sulfur hexafluoride. Inject the propellant sample to be tested into the column in the same manner as the calibration standard. Record the retention times and the peak areas. Repeat two times.

4.5.2.4 Calculation. Compare the average peak area of the calibration standard to that of the nitrogen trifluoride sample being tested. Calculate the concentration of each component using the formula below:

$$\frac{Sample\ Peak\ Area}{Standard\ Peak\ Area} \times (Std\ Conc) = Component\ Concentration$$

4.5.3 Oxygen + argon, nitrogen, and carbon monoxide. The concentration of oxygen+argon (O_2+Ar), nitrogen (N_2), and carbon monoxide (CO) in the propellant shall be determined by gas chromatography.

4.5.3.1 Instrument parameters. The analyst may vary the column dimensions, carrier gas flow rate, and temperatures to optimize the analysis.

Detector:

Thermal conductivity detector, discharge ionization detector, or a pulsed discharged helium ionization detector.

Column:

Packing:	Molecular sieve 13X, 80/100 (or equivalent)
Length:	6.1 m (20 ft)
ID:	1.75 mm (1/16 in.)
OD:	3.2 mm (1/8 in.)
Column material:	stainless steel

Carrier Gas / Flow Rate:

Helium (MIL-PRF-27407) / 17 mL/min

Temperatures:

Detector:	140°C
Filament:	240°C
Oven:	100°C
Gas sampling valve:	60°C

4.5.3.2 Calibration. Prepare a gaseous standard of helium containing at least 0.1% NF_3 and the components being analyzed in a concentration between one tenth and 10 times the specification limit or of the predicted measurement in the propellant sample. Analysis of the calibration standard must show a clear separation of each specified component from all other components and from NF_3.

4.5.3.3 Procedure. Inject the calibration standard into the column using a gas-sampling valve. Record the retention times and peak areas for each component. The order of elution is oxygen+argon, nitrogen, carbon monoxide, and nitrogen trifluoride. Inject the propellant sample to be tested into the column in the same manner as the calibration standard. Record the retention times and the peak areas. Repeat two times.

4.5.3.4 Calculation. Compare the average peak area of the calibration standard to that of the nitrogen trifluoride sample being tested. Calculate the concentration of each component using the formula in 4.5.2.4.

4.5.4 Total reactive fluoride as HF. The concentration of total reactive fluoride as HF in nitrogen trifluoride is determined by one of the following procedures. In the event of dispute the procedure outlined in 4.5.4.1 shall be the referee method.

4.5.4.1 Fluoride ion selective electrode.

4.5.4.1.1 Buffer solution. Place 500 mL of distilled or de-ionized water in a 1000 mL volumetric flask. Add 57 mL glacial acetic acid and 58 g of sodium chloride. Adjust the pH to between 5.0 - 5.5 with 5 M sodium hydroxide. Cool to room temperature. Dilute to the mark with distilled or de-ionized water.

4.5.4.1.2 Calibration standard. Sodium fluoride (10^{-3} M F^- in water, freshly prepared).

4.5.4.1.3 Procedure.

4.5.4.1.3.1 Calibration curve. Prepare a working standard by adding 100 mL of the 10^{-3} M F^- solution to 100 mL of buffer solution. Prepare a blank containing 50 mL of 0.2 N NaOH and 50.0 mL buffer solution in a plastic beaker. Place a reference electrode (single junction type) and a fluoride ion selective electrode, connected to a millivolt meter (0.1 mV scale), into the blank. Gently stir the blank and record the mV reading when the reading becomes stable. Successively add increments of the working standard to the blank to generate a calibration curve. Record stable mV reading after each addition.

4.5.4.1.3.2 Sample preparation. Place 50 mL of 0.2 N NaOH solution into each of two PTFE bubblers connected in series. Establish a flow of < 1000 sccm of the propellant through the bubblers using a suitable flow controller or flow meter. Continue the flow of propellant through the bubblers until approximately 15 liters (at STP) of the propellant has been sparged through the 0.2 N NaOH solution. Transfer the contents of each bubbler to separate 100 mL volumetric flasks and add 50 mL of the buffer solution to each. If necessary, fill to the mark with de-ionized or distilled water.

4.5.4.1.3.3 Measurement. Transfer the contents of the volumetric flasks prepared in 4.5.4.1.3.2 into separate plastic beakers. Place the electrodes into each beaker and record the mV readings for each in the same manner as for the blank. Determine the F^- concentration using the calibration curve generated in 4.5.4.1.3.1.

4.5.4.1.3.4 Calculation. Calculate the concentration of total reactive fluorides as HF in the propellant using the equation below. The HF concentration of the second bubbler should be insignificant compared to the first bubbler. If significant HF levels are found in the second bubbler, prepare a new sample with a lower flow rate.

$$Total\ Reactive\ Fluorides\ as\ HF\ (Vol\%) = C \times 0.11 \times \left(\frac{22.4\ L/mole}{Vs}\right) \times 100$$

where,

C = F^- concentration (M) determined in 4.5.4.1.3.3.

Vs = Volume of propellant sampled (liters at STP).

4.5.4.2 Fourier Transform Infrared Spectroscopy (FTIR). Determine the HF content on an FTIR spectrometer that has been validated and calibrated for an HF concentration of approximately 0.05% vol.

4.5.5 Water. The concentration of water in the propellant is determined by one of the following methods. In case of dispute the electrolytic method in 4.5.5.1 shall be the referee method.

4.5.5.1 Electrolytic. Connect the sample container to a pressure regulator that is attached to an electrolytic moisture apparatus (hygrometer). Open the sample container valve and adjust the pressure to the apparatus in accordance with the manufacturer's recommended value. Allow sufficient time for the indicated moisture content to become stable and read the value obtained while using the most sensitive scale setting possible for the moisture content of the sample. The hygrometer should be set on a range no greater than ten times the specified maximum moisture content.

4.5.5.2 Piezoelectric. Determine the moisture content utilizing a piezoelectric sorption hygrometer that is set on a range no greater than ten times the specified maximum moisture content by following the instrument manufacturer's instructions.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Service or Defense Agency, or within the military service's system commands. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended Use. The propellant covered by this purchase description is intended for use as an oxidizer in various reactant systems.

6.2 Acquisition requirements. Acquisition documents must specify the following:

a. Title, number, and date of this specification.

b. Method of shipment, type and capacity of containers.

c. When variations in points of inspection are granted (4.1).

d. When variation in quality conformance tests is granted (4.3)

e. When variation in sampling method is granted (4.4)

f. When cleaning and repair is required for lease or Government owned containers (5.1.1).

6.3 Packaging requirements. Guidance for cylinders may be found in the following documents:

MIL-STD-101	-	Color Code for Pipelines and for Compressed Gas Cylinders
RR-C-901	-	Cylinders, Compressed Gas: Seamless Shatterproof, High Pressure DOT 3AA Steel, and 3AL Aluminum
49 CFR 171 - 181	-	Hazardous Materials Regulations
CGA C-6	-	Standards for Visual Inspection of Steel Compressed Gas Cylinders
CGA C-6.1	-	Standards for Visual Inspection for High Pressure Aluminum Compressed Gas Cylinders

6.4 Subject term (key word) listing

Oxidizer

Electrolytic

Piezoelectric

6.5 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

CONCLUDING MATERIAL

Custodians:
Army - MI
Navy – AS
Air Force – 68
DLA – PS

Preparing activity:
Air Force – 68
(Project 9135-2010-001)

Review activities:
Air Force – 19

Note: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information using the ASSIST Online database at https://assist.daps.dla.mil.