

MIL-P-87896
4 OCT 1982

MILITARY SPECIFICATION

Propellant, Nitrogen Trifluoride

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

1. SCOPE

1.1 Scope. This specification covers the requirements for nitrogen trifluoride (NF₃).

1.2 Classification. Nitrogen Trifluoride shall be of the following types as specified (6.2).

Type I. - Gaseous

2. APPLICABLE DOCUMENTS

2.1 Government Documents

2.1.1 Specifications and Standards. The following specifications and standards of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DODISS) specified in the solicitation, form a part of this specification to the extent specified herein (6.2).

SPECIFICATIONS

Military

MIL-C-5586	- Cylinder, Oxygen, Low Pressure, Nonshatterable
MIL-C-81302	- Cleaning Compound, Solvent, Trichlorotrifluoroethane
MIL-C-83690	- Cylinders, Sampling
MIL-P-27401	- Propellant, Pressurizing Agent Nitrogen
MIL-P-27407	- Propellant, Pressurizing Agent Helium

STANDARDS

Military

MIL-STD-1411	- Inspection and Maintenance of Compressed Gas Cylinders
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FSC 9135

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

2.1.2 Other Government Documents. The following other Government documents form a part of this specification to the extent specified.

DEPARTMENT OF TRANSPORTATION (DOT)

49 CFR 170-179 - Department of Transportation Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles.

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

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 investigation for bids or request for proposals shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

E 29	- Standard Recommended Practice for Indicating Which Places of Figures are to be Considered Significant in Specified Limiting Values
F 307	- Pressurant Gas Sampling for Gaseous Analysis

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

3. REQUIREMENTS

3.1 Composition. The composition of the NF_3 shall conform to the limits in Table I when tested in accordance with the applicable test methods (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 place of figures used in expressing the limitation value, in accordance with ASTM Method E29.

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TABLE I, COMPOSITION

COMPOSITION	LIMIT	TEST PARAGRAPH
Assay, (purity), Nitrogen Trifluoride (NF ₃) percent by vol, min	98.0	4.5.4
Tetrafluoromethane (CF ₄) percent by vol, max	1.0	4.5.2
Carbon Monoxide (CO)/Oxygen (O ₂) + Nitrogen (N ₂), percent by vol, max	0.5	4.5.2
Carbon Dioxide (CO ₂), percent by vol, max	0.1	4.5.2
Nitrous Oxide (N ₂ O), percent by vol, max	0.2	4.5.2
Difluorodiazine (N ₂ F ₂), percent by vol, max	0.1	4.5.2
Total Reactive Fluoride (as HF, hydrogen fluoride), percent by wt, max	0.1	4.5.1
Water (H ₂ O), parts per million (ppm) by vol	5	4.5.3

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 purchase order, the supplier may use his own or any other facilities suitable for their 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 this specification where such inspections are deemed necessary to assure supplier and services conform to the prescribed requirements (6.2).

4.1.1 Point of Inspection. Unless otherwise specified, quality conformance tests of the propellant shall be conducted at the site of filling. The quality conformance tests shall be completed prior to shipment of filled containers (6.2).

4.2 Classification of Tests. The inspection and testing of NF₃ shall be classified as quality conformance tests.

4.3 Quality Conformance Tests. Quality conformance tests are defined as tests specified herein to assure conformance to the requirements of Table I and MIL-STD-1411.

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4.3.1 Individual Tests. Each Type I NF₃ cylinder shall be subjected to the following tests as specified in MIL-STD-1411:

4.3.1.2 Leakage

4.3.2 Sampling Tests. The number of NF₃ containers shall be selected in accordance with Table II and subjected to the tests described under 4.5.

TABLE II, SAMPLING FOR TEST

<u>NUMBER OF CONTAINERS IN LOT</u>	<u>NUMBER OF CONTAINERS TO BE SAMPLED</u>
1	1
2-40	2
41-70	3
70-over	4

4.3.2.1 Lot Definitions.

4.3.2.1.1 The NF₃ 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.

4.3.2.1.2 The NF₃ 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.

4.3.2.1.3 The NF₃ from either of 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.

4.3.2.2 Sample. Each sample shall be of sufficient size to conduct all the quality conformance tests as specified herein. Unless otherwise specified, the quality conformance tests shall be made of each required sample (6.2). When required, an equivalent sample shall be forwarded to a laboratory designated by the procuring activity for subjection to the quality conformance tests specified herein.

4.3.2.2.1 Samplers. The sampler for (gaseous) NF₃ shall be in accordance with MIL-C-83690 or functionally equivalent thereto. The aliquots taken for analysis are representative samples. Passivation of the samplers is to be achieved by slowly bleeding in NF₃ after cleaning, valve assembly, nitrogen purge, and leak testing.

4.3.2.2.2 Sampling Methods. Unless otherwise specified, Type I (gaseous) NF₃ shall be sampled in accordance with ASTM F307. All apparatus used shall be made of suitable materials.

4.3.3 Cylinders. The number of cylinders (pressure and Dewar types) filled with Type I NF₃ selected for sampling for each lot shall be in accordance with Table II. The first and last cylinders to be filled within a given lot shall be sampled. Other samples may be selected at random. The NF₃ from each cylinder sampled shall constitute a separate sample. For the purposes of selecting sample cylinders only, any one cylinder may be selected from a group of cylinders filled simultaneously from a single manifold.

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

4.5 Test Methods.

4.5.1 Total Reactive Fluorides. Total reactive fluorides as HF, shall be determined as follows:

4.5.1.1 Apparatus. Flush the individual components with trichlorotrifluoroethane and purge dry with nitrogen. Assemble the apparatus as shown in Figure I. Evacuate the sample reservoir and gas washing bottle to less than 1mm pressure and insure that no leak exists (no pressure change for 30 minutes).

4.5.1.2 Procedure. Locate the apparatus in a fume hood. Disassemble the gas washing bottle, transfer 40 ml of 0.1N-NaOH into the glass vessel, and reassemble the apparatus. Connect the apparatus to the NF₃ sample source through a regulator and/or metering valve (A) with valve (B) closed evacuate the sample reservoir through valve (C) to 0 mm pressure indicated by the gauge. Close valve (C) and slowly open valve (B). Record the pressure (P_i). Open valve (A) slowly so that sample is admitted without sparging liquid out of the gas washing bottle. Fill the sample reservoir with NF₃ to approximately 760 mm and close valve (A). Record the pressure (P_f). Close valve (B) and remove the glass vessel from the gas washing bottle. Disconnect the apparatus from the NF₃ source and allow any liquid trapped in the polytetrafluoroethylene tube to drain into the glass vessel. Transfer the liquid to a beaker and add 40 ml of T.I.S.A.B. solution to the mixture. After mixing the solution, determine the fluoride ion concentration using the fluoride selective ion electrode and calibration plot. Record the fluoride ion concentration (ppmF).

4.5.1.3 Calculation. Calculate the total reactive fluoride as follows:

$$\text{wt\%HF} = \frac{\text{ppmF} \times 80 \times 1.05 \times 22.4 \times 760 \times 273}{V \times (P_f - P_i) \times T \times 71 \times 10^4} = \frac{\text{ppmF} \times 550}{V \times (P_f - P_i) \times T}$$

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where: ppmF = parts per million fluoride ion in the 80 ml solution

P_f = final pressure (mm)

P_i = initial pressure (mm)

T = ambient temperature ($^{\circ}$ K)

V = volume of the sample reservoir (liters)

4.5.1.4 Electrode Calibration. Dilute 10, 50, and 100 ml of 1000 ppm fluoride solution contained in separate 1000 ml volumetric flasks to the mark with 50/50 T.I.S.A.B./water solution. Record the millivolt reading produced by the fluoride electrode with each solution (10, 50 and 100 ppmF). Plot log concentration versus millivolts.

4.5.1.5 Reagents and Equipment. The following apply as test conditions of 4.5.1.

(a) Reagents.

1. Trichlorotrifluoroethane, conforming to MIL-C-81302.
2. Gaseous Nitrogen, conforming to MIL-P-27401.
3. O.IN-NaOH, 4g ACS reagent grade NaOH dissolved in distilled water and diluted to one liter. Store in a polyethylene container.
4. T.I.S.A.B., T.I.S.A.B.II, Orion catalog Number 940909, or equivalent.
5. 1000 ppmF solution, 2.21g. dry ACS reagent grade NaF dissolved in water and diluted to one liter. Store in a polyethylene container.

(b) Equipment.

1. Specific Ion Meter, Orion Model 401, or equivalent.
2. Fluoride Electrode, Orion Cat. No. 940900, or equivalent.
3. Reference Electrode, single junction, Orion Cat. No. 900100, or equivalent.
4. Cylinder, 280 cu. (4.588l) internal volume, Type B-3, MIL-C-5586, or equivalent.
5. Valves, Diaphragm seal, Hoke Type 413, or equivalent.
6. Gauge, absolute pressure, 0-760 mm, Matheson part no. 63-5601, or equivalent.

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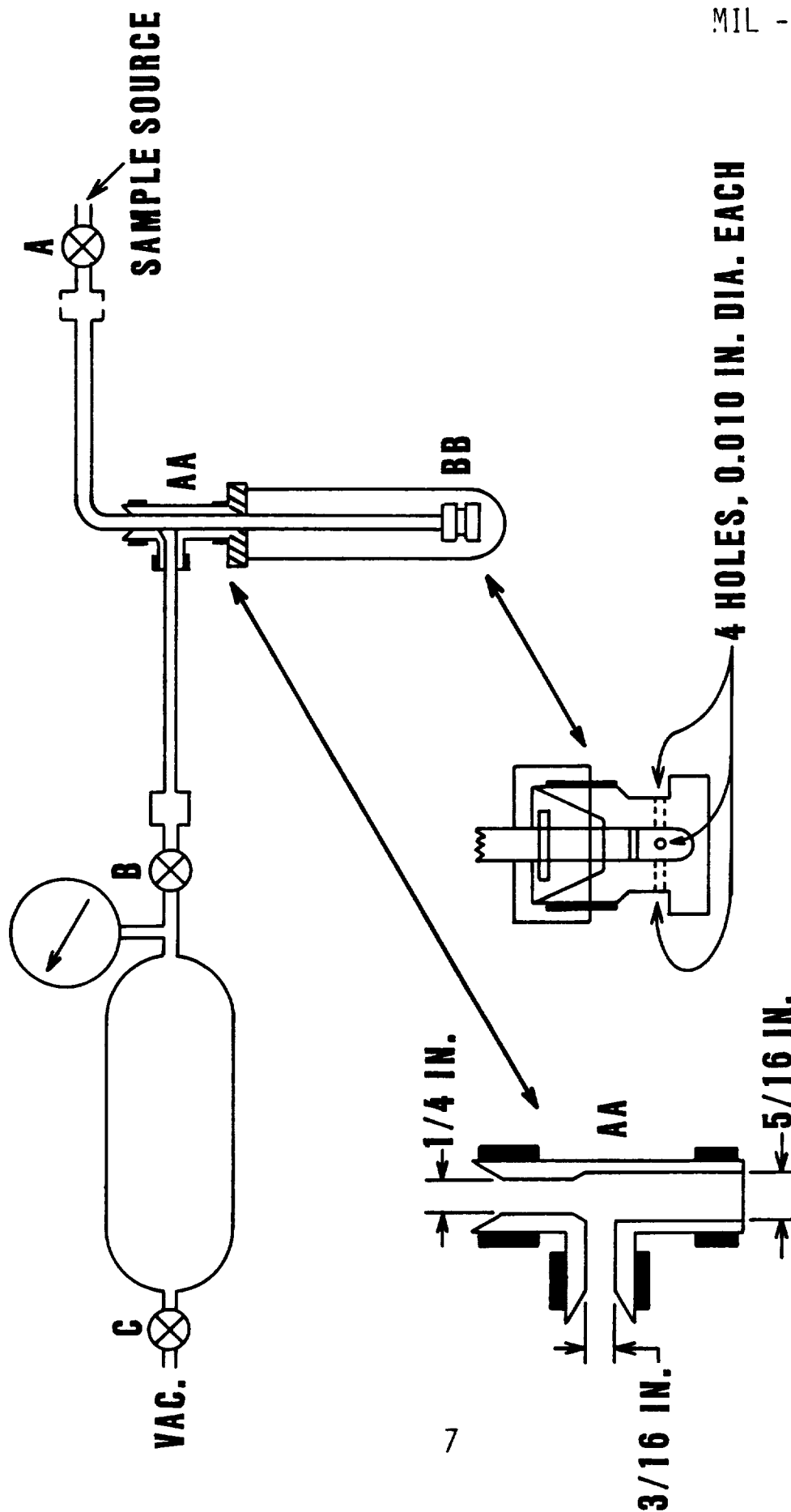


Figure 1, TOTAL FLUORIDE APPARATUS

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7. Aerosol compatability tube, 100 ml volume, Fisher-Porter or equivalent.
8. "Tee" Fitting, Swagelok 400 3TMT-316, drilled as shown, or equivalent.
9. Cap - Swagelok 400-C-Teflon, drilled as shown, or equivalent.
10. Tubing, 1/4 in O.D. x 1/8 in I.D., Polytetrafluoroethylene or Polychlorotrifluoroethylene.
11. Pump, vacuum, capable of 0.1 mm Hg or less.

4.5.2 Gaseous Impurities. Gaseous impurities shall be determined by gas chromatography as follows:

4.5.2.1 Apparatus. Fill a 20 ft x 1/8 in O.D. stainless steel tube with 80/100 mesh Chromosorb 102. Mechanically vibrate or tap the tube to insure uniform packing and plug the ends of the prepared gas chromatograph column with glass wool. Fit the column into the chromatograph oven and connect one end to the sample inlet, without connecting the other end to the detector, heat the oven to 200°C and condition the column for 4 hours with carrier gas flowing. After conditioning the column, connect the other end to the thermal conductivity detector and adjust the carrier gas flow to approximately 15ml/min. Set the column oven to 35° C and the detector current to a nominal sensitivity for helium carrier gas. Locate the sample exhaust tube of the gas sampling valve in a fume hood with the end of the tube in the flame from a Bunsen burner.

4.5.2.2 Procedure. Slowly open the source valve until the exhaust end of the gas sampling valve outlet indicates NF₃ is passing through the system. Allow the system to purge for 10-20 seconds and then turn off the NF₃ source. Immediately inject the sample into the gas chromatograph. Record the areas of all peaks in the chromatogram. Figure 2 shows a typical chromatogram of NF₃ and one of the selected impurities which may be found in the sample. The component elution order is H₂, N₂, CO(O₂), NO, CF₄, NF₃, N₂F₂, CO₂, N₂O and SF₆. NO₂ may appear as a tailing peak between NO and CF₄.

4.5.2.3 Calculation. Calculate the concentration of the following gaseous impurities: N₂, CO(O₂), NO, CF₄, N₂O, SF₆, CO₂ and trans-N₂F₂ as follows.

$$\text{Vol \%} = A \times S / A$$

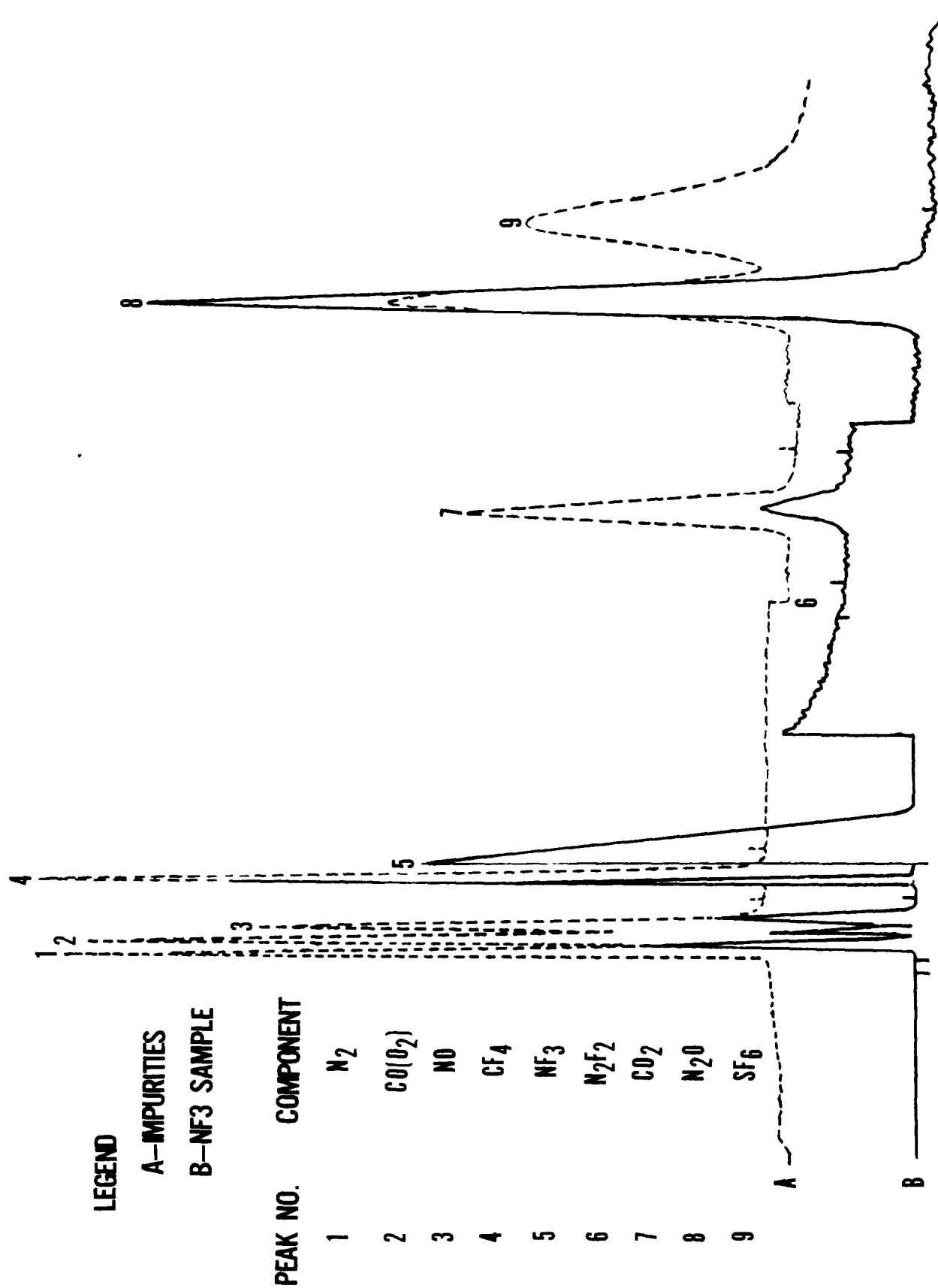
where: A = the area of the specific impurity peak.

S = the corresponding sensitivity factor of the impurity.

A = sum of the areas

4.5.2.4 Calibration. Use the procedure described in 4.5.2.2 and prepare gaseous standards containing approximately 0.1% each N₂, CO, NO, CF₄, CO₂, N₂O, and SF₆ in Helium. Calculate the sensitivity of each component as follows.

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Figure 2, GAS CHROMATOGRAMS OF IMPURITIES AND A TYPICAL NF₃ SAMPLE

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$$S = \frac{\%}{A_s}$$

where: % = Vol percent of specific impurity present.
 A_s = corresponding area of the specific impurity peak.

4.5.2.5 Reagents and Equipment. The following apply as test conditions of 4.5.2.

(a) Reagents.

1. Helium, corresponding to MIL-P-27407.
2. Chromosorb 102, 80/100 mesh, Johns-Manville Co., or equivalent.
3. Sodium thiosulfate pentahydrate, ACS reagent grade, 30/50 mesh.
4. Calibration Standards, 0.1% N_2 , CO, NO, CF_4 , CO_2 , N_2O and SF_6 in Helium.

(b) Equipment.

1. Tubing, stainless steel, 20 ft x 1/8 in O.D.
2. Tubing, glass or stainless steel 8 in x 1/4 in O.D.
3. Gas Chromatograph, equipped with a thermal conductivity detector and a gas sampling valve (1 ml loop volume).
4. Integrator, electronic. Capable of recording retention time and area.
5. Bunsen burner.
6. Recorder, potentiometric, 1 sec. F.S., 1 mv span, (Optional).

4.5.3 The Water Content. The water content of the sample shall be determined by one of the following methods. In cases of dispute the electrolytic method in 4.5.3.1 shall be used as the referee.

4.5.3.1 Electrolytic Method. Connect the sample container to a pressure regulator which is attached to the 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 electrolytic moisture apparatus should be set on a range no greater than ten times the specified maximum moisture content.

4.5.3.2 Frost (Dew) Point Method.

4.5.3.3 By a piezoelectric sorption hygrometer that is set on a range no greater than ten times the specified maximum moisture content.

4.5.3.4 By an aluminum oxide capacitor-equipped analyzer on a range that is no greater than ten times the specified maximum moisture content.

4.5.4 Final Calculation. Calculate the NF₃ content of the sample as follows:

$$\text{Vol \%NF}_3 = 100 - \sum \%i$$

where: $\sum \%i$ = the sum of the impurities found in 4.5.1, 4.5.2 and 4.5.3.

5. PREPARATION FOR DELIVERY

5.1 Gas Cylinders. Type I NF₃ cylinders shall be prepared in accordance with procedures for oxygen in MIL-STD-1411 with the following exceptions:

5.1.1 The odor test is omitted.

5.1.2 The cylinder and tube valve outlets shall be according to Compressed Gas Association Valve Outlet 330. The main manifold valve on the tube tank trailers shall be Circle Seal Valve P/N MV 3308P/MV 3308P-P unless otherwise specified (6.2).

5.1.3 Each cylinder shall be color coded and marked according to MIL-STD-101 as follows:

<u>TOP A</u>	<u>BAND B</u>	<u>BAND C</u>	<u>BODY</u>
Brown	Green	Green	Brown

5.2 Identification Tag. Unless otherwise specified in the contract or purchase order, and identification tag impervious to climatic conditions shall be wired to the outlet port of each container and shall contain the following information: Product name, specification number with revision letter, NSN, quantity, name of manufacturer, name of contractor (if different from manufacturer), date of manufacture, and lot identification number (6.2).

6. NOTES

6.1 Intended Use. The nitrogen trifluoride is used as an oxidizer in various reactant systems.

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

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6.2.1 Procurement Requirements.

- (a) Title, number, type and date of this specification.
- (b) When inspection requirements are to be performed by other than the supplier (4.1).
- (c) Method of shipment, type and capacity of container (6.3).
- (d) When variation in points of inspection are granted (4.1.1).
- (e) When waiver on quality conformance tests on each sample is granted (4.3.2.2).
- (f) When variation in sampling method is granted (4.3.2.2.2).
- (g) When disposition of rejected product has to be specified (4.4).
- (h) When instructions for disposition of rejected cylinders are required (MIL-STD-1411).
- (i) When cleaning and repair provisions are other than specified (MIL-STD-1411 and 5.2.1).
- (j) When approval of gasket material is required (5.2.2).
- (k) When identification tag is to be other than specified (MIL-STD-1411).
- (l) When container color code is waived (MIL-STD-1411 and 5.1.3).
- (m) When variation in requirement is granted (3.1).
- (n) When other values are acceptable (5.1.2).
- (o) When identification tag is to be other than specified (5.2).

6.2.2 Contract Data Requirements. Data conforming to Data Item Description DI-T-3773, Quality Conformance Test Reports, is a requirement for delivery in connection with this specification. The data items will be specified for delivery on the DD 1423.

6.3 Containers. As of the date of this specification, the following listed containers are considered acceptable for military use and approved for NF₃ as specified by DOT.

6.3.1 Type I (Gaseous).

6.3.1.1 Cylinders of specification DOT 3A, 3AA, 3AAX, and 3E1800.

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6.4 Field Use Limits. The requirements established by this specification are applicable for procurement purposes only and are valid solely as utilized by vendor and the procuring activity. They are not intended to constitute field use limits, which should be determined for each user application.

Custodians:

Army - MI

Navy - AS

Air Force - I2

Review Activities:

Army - MI

Navy - AS

Preparing Activity:

Air Force - I2

Other Agency
Interest:

NASA

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F081

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