### METRIC

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SUPERSEDING MIL-PRF-7254G 15 December 1997

# **PERFORMANCE SPECIFICATION**

# **PROPELLANT, NITRIC ACID**



Comments, suggestions, or questions on this document should be addressed to AFPET/PTPS 2430 C Street, Bldg. 70, Area B, Wright-Patterson AFB OH 45433-7632 or e-mailed to <u>AFPA.PTPS@us.af.mil.</u> Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <u>https://assist.dla.mil</u>.

AMSC N/A

FSC 9135

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.

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

1. SCOPE

- 1.1 <u>Scope</u>. This specification covers the requirements for four types of nitric acid propellant.
- 1.2 <u>Classification</u>. The nitric acid will be of the following designated types:

Type IIIA	-	Nominal 14 percent NO <sub>2</sub> 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.
Type IV	-	High density propellant with nominal 44 percent NO <sub>2</sub> content, lower solids content, plus corrosion inhibitor and limited iron content.

1.3 <u>Part or identifying number (PIN)</u>. The PINs to be used for nitric acid acquired to this specification are created as follows:

M	<u>7254</u> – <u>X</u>	Example of PIN:	M7254-3B
		Type (see 1.2). Use "3 type IIILS; "4" for type I	BA" for type IIIA; "3B" for type IIIB; "3LS" for IV.
		Specification number	
		Prefix for military speci	fication

## 2. APPLICABLE DOCUMENTS

2.1 <u>General</u>. The documents listed in this section are specified in sections 3 and 4 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 and 4 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 <u>Specifications, standards, and handbooks</u>. 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-27401 Propellant Pressurizing Agent, Nitrogen

(Copies of these documents are available online at http://quicksearch.dla.mil.)

2.3 <u>Non-government publications</u>. 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 D1068	Standard Test Methods for Iron in Water (DoD adopted)
ASTM E29	Standard Practice for Using Significant Digits in Test Data to
	Determine Conformance with Specifications (DoD adopted)

(Copies of these documents are available from http://www.astm.org.)

2.4 <u>Order of precedence</u>. Unless otherwise noted herein or in the contract, in the event of a conflict between the text of this document and the references cited herein (except for related specification sheets), 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 <u>Chemical and physical properties</u>. The chemical and physical properties of the propellant shall conform to those listed in Table I when tested in accordance with the applicable test methods.

Properties	Type IIIA	Type IIIB	Type IIILS	Type IV	Test Paragraph
HNO <sub>3</sub> (% by wt)	81.6 - 84.9	81.7 – 84.9	83.7 – 86.4	52.7 – 57.4	4.3.2
NO <sub>2</sub> (% by wt)	14 ± 1	14 ± 1	14 ± 1	44 ± 2	4.3.4
HF (% by wt)	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.1	4.3.5
H <sub>2</sub> O (% by wt)	2.0 ± 0.5	2.0 ± 0.5	0.5 max	0.5 max	4.3.6
Fe <sub>2</sub> O <sub>3</sub> (% by wt)	Not specified	Not specified	0.0015 max	0.002 max	4.3.7
Solids (% by wt as nitrates), max	0.10	0.04	0.04	0.04	4.3.8
Relative density @ 15.6 °C/15.6 °C, g/mL	1.564 – 1.575	1.564 – 1.575	1.572 – 1.582	1.642 – 1.652	4.3.9

TABLE I. Chemical and	physical	properties.
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3.2 <u>Limiting values</u>. 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 Method of ASTM Practice E29.

3.3 <u>Qualitative</u>. The propellant shall be a clear, single-phased liquid when examined visually by transmitted light and the absorbance spectrum resulting from the water analysis (Appendix D) shall contain the 1440 nm peak which identifies the presence of nitric acid.

## 4. VERIFICATION

4.1 <u>Classification for inspections</u>. The inspections shall be classified as quality conformance inspections.

4.2 <u>Quality conformance inspection</u>. The propellant shall be sampled. Each sample shall be subjected to the visual examination described in 4.3.1 for conformance to 3.3 and to the tests specified in Table I.

#### 4.2.1 Sampling plan.

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 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 that is used to fill shipping containers directly from the process output. A batch process shall be the production of product by single additions of raw materials that 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.

4.2.1.2 <u>Sample size</u>. A sample consists of not less than 150 milliliters (mL) of propellant. Unless otherwise specified, the supplier is responsible for the performance of all inspection requirements as specified herein. When required, the sample shall be forwarded to a laboratory designated by the procuring activity for subjection to the quality conformance tests specified.

4.2.1.2.1 <u>Containers of 400 liters or less water capacity</u>. The number of containers 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 container 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.

4.2.1.2.2 <u>Containers greater than 400 liters water capacity</u>. Each container shall constitute a lot. Unless otherwise specified, the sample shall be from a point 6 to 12 inches from the bottom of the shipping container and shall be tested to determine compliance with this specification.

Number of Containers in Lot	Number of Containers Sampled
1	1
2 – 40	2
41 – 70	3
71 and over	4

### TABLE II. Sampling for tests.

4.2.2 <u>Rejection</u>. When any sample of the propellant tested in accordance with 4.3 fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected.

4.3 <u>Test methods</u>.

4.3.1 <u>Examination of product</u>. The propellant shall be visually examined to ensure that the material conforms to 3.3. The examination shall be conducted after a 25 mL aliquot of the propellant sample has been transferred to a polychlorotrifluoroethylene (PCTFE) vessel or equivalent.

4.3.2 <u>Nitric acid</u>. The HNO<sub>3</sub> content of the propellant sample shall be determined by subtracting the components analyzed from 100 as follows:

 $HNO_3 Wt\% = 100 - (\%NO_2 + \%HF + \%H_2O + \%solids)$ 

4.3.3 <u>Neutralization</u>. The propellant sample shall be neutralized by the procedure described in Appendix A.

4.3.4 <u>Nitrogen dioxide</u>. The nitrogen dioxide of the propellant sample shall be determined by the procedure described in Appendix B.

4.3.5 <u>Hydrogen fluoride</u>. The hydrogen fluoride content of the propellant sample shall be determined by the procedure described in Appendix C.

4.3.6 <u>Water</u>. The water content of the propellant sample shall be determined by the procedure described in Appendix D.

4.3.7 <u>Iron</u>. The iron content as  $Fe_2O_3$  of the propellant sample shall be determined by the procedure described in Appendix E (referee) or by ASTM D1068, Test Method A (Atomic Absorption, Direct), paragraph 12 (Standardization).

4.3.8 <u>Solids</u>. The solid content of the propellant sample shall be determined by the procedure described in Appendix F.

4.3.9 <u>Relative density</u>. The relative density of the propellant sample shall be determined at 15.6 °C/15.6 °C (60 °F/60 °F) by any convenient standard method, such as a hydrometer, and shall conform to the limits in Table I. (Note : Because of HF attack, periodic calibration will be required).

## 5. PACKAGING

5.1 <u>Packaging</u>. 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 <u>Intended use</u>. The propellant described by this specification is intended for use as a fuel 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.

#### 6.2 <u>Acquisition requirements</u>. Acquisition documents must specify the following:

- a. Title, number, and date of this specification.
- b. Type and capacity of containers.
- c. Quantity by weight in pounds.
- d. When a different sampling plan is required (4.2).
- e. Packaging requirements (5.1).

6.3 <u>Safety Data Sheets</u>. Contracting officers will identify those activities that require copies of completed Safety Data Sheets (SDS) prepared in accordance with the 29 CFR 1910.1200.

- 6.4 Subject term (key word) listing.
  - Acetonitrile Fuel Hydrogen fluoride Inhibitor Sulfuric acid

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

## MIL-PRF-7254H APPENDIX A

#### NEUTRALIZATION

A.1 SCOPE

A.1.1 <u>Scope</u>. This Appendix describes a procedure for the neutralization of the propellant sample. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

## A.2 PROCEDURE

A.2.1 <u>Sample preparation.</u> Transfer 1 mL of the propellant sample 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 about 1.3 grams (g) when using 0.5 Normal (N) base.

A.2.1.1 <u>Procedure</u>. Immerse the weighed vial in a bath of powdered carbon dioxide (dry ice) or liquid nitrogen and freeze. Concurrently, add a measured amount of 0.5 N sodium hydroxide (NaOH) which is in excess of that required for neutralization (the amount of base needed shall be calculated after the sample weight is known) to a 500 mL heavy- walled, glass stoppered iodine flask. Place the flask in a salt water ice bath for 5 minutes (min). Displace the air in the flask with gaseous nitrogen conforming to MIL-PRF-27401 and insert the glass stopper immediately after. Open the top of the frozen vial, drop it into the iodine flask, and immediately seal with the glass stopper. Hold the stopper in place and shake the flask vigorously. Allow the flask to stand for 15 min at room temperature with occasional shaking during this time. A small amount of purified water shall be poured into the lip of the flask, with the stopper loosened to allow the water to run into the flask. The stopper shall be rinsed with a stream of purified water, also into the flask. Add 3 to 5 drops of phenolphthalein indicator to ensure that the contents of the flask are slightly alkaline with respect to phenolphthalein.

A.2.1.2 <u>Reagents and equipment</u>. The following shall apply as test conditions.

A.2.1.2.1 <u>Reagents</u>. Commercially available analytical standards of suitable purity may be used where applicable.

a. Sodium hydroxide solution (NaOH), 0.5 N: Dissolve 20 g of American Chemical Society (ACS) reagent grade, low carbonate, NaOH in purified water in a 1 liter (L) volumetric flask. Allow the solution to cool and dilute to the mark with purified water. Store in a polyethylene vessel and ensure the exclusion of atmospheric carbon dioxide.

- b. Phenolphthalein indicator.
- c. Gaseous nitrogen conforming to MIL-PRF-27401.

#### A.2.1.2.2 Equipment.

- a. Flask, iodine, heavy- walled glass stoppered, 500 mL.
- b. Vials, polyethylene or polypropylene.
- c. Flask, volumetric, 1 L.
- d. Salt water ice bath.
- e. Powdered carbon dioxide (dry ice) or liquid nitrogen bath.

## MIL-PRF-7254H APPENDIX B

#### NITROGEN DIOXIDE

#### B.1 SCOPE

B.1.1 <u>Scope</u>. This Appendix describes a method for the determination of the  $NO_2$  content in the propellant. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

#### B.2 METHOD

B.2.1 <u>Procedure</u>. The slightly alkaline sample of 4.3.3 & Appendix A shall be transferred quantitatively to a 250 mL volumetric flask and diluted to volume with purified water. A 50 mL aliquot of this solution shall be withdrawn, 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 4.3.5 & Appendix C. One mL of 1 N sulfuric acid ( $H_2SO_4$ ) shall be added with stirring. Add without stirring an exact volume of 0.1 N ceric solution sufficient to react the nitrite ion (the amount can be calculated from the aliquot weight of the sample, plus 5 mL in excess). The beaker shall be placed on the stirring platform with the electrode assembly immersed and a blanket of gaseous nitrogen conforming to MIL-PRF-27401 covering 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. Determine the end point by plotting the volume (mL) versus millivolts (mV).

B.2.2 <u>Calculation</u>. Use the following formula to calculate the NO<sub>2</sub> content of the propellant:

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

Where:

N = Normality of the ceric solution.

 $N_1$  = Normality of the ferrous ammonium sulfate.

W = Original propellant sample weight in (g).

#### B.2.3 Reagents and equipment.

B.2.3.1 <u>Reagents</u>. Commercially available analytical standards of suitable purity may be used where applicable.

a. Standard ceric solution 0.1 N: Dissolve exactly 54.878 g of dry certified ACS reagent grade ceric ammonium hexanitrate  $[(NH_4)_2Ce(NO_3)_6]$  in 2 N  $H_2SO_4$  in a 1 L volumetric flask dilute to the mark with 2 N  $H_2SO_4$ . The ceric nitrate is dried for two hours at 60 °C (140 °F) in a vacuum oven. Store the solution in a polyethylene bottle with a gaseous nitrogen atmosphere. A solution which has been prepared in excess of seven days ahead of the analysis shall be standardized weekly before use with NIST primary standard grade arsenic trioxide using ferroin as the indicator.

b. Standard ferrous solution, 0.05 N: Dissolve 19.608 g of ACS reagent grade ferrous ammonium sulfate hexahydrate, [Fe(NH<sub>4</sub>)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>•6H<sub>2</sub>O] in 0.5 N H<sub>2</sub>SO<sub>4</sub> in a 1 L volumetric flask, and

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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 B.2.1.

c. Sulfuric acid solution, 0.5, 1 and 2 N: Dilute 14, 28, and 56 mL, respectively, of concentrated ACS reagent-grade  $H_2SO_4$  to 1 L with purified water.

d. Gaseous nitrogen conforming to MIL-PRF-27401.

B.2.3.2 Equipment.

- a. Ice bath, salt water.
- b. Electrode assembly, platinum and saturated calomel or suitable alternative.
- c. pH meter with millivolt scale.

## MIL-PRF-7254H APPENDIX C

### HYDROGEN FLUORIDE

C.1 SCOPE

C.1.1 <u>Scope</u>. This Appendix describes a method for the determination of the HF content in the propellant. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

#### C.2 METHOD

C.2.1. <u>Procedure</u>. Transfer a 100 mL aliquot of the slightly alkaline solution of B.2.1 to a 500 mL volumetric flask, dilute to the mark with purified water, and mix. Immediately transfer a selected volume (25 to 50 mL) to a plastic 100 to 200 mL beaker. Adjust the pH to 5.5 by the addition of an equal volume of citrate buffer. Check the calibration curve from C.2.2 using the 2 and 4  $\mu$ g/mL (ppmv) fluoride standard solutions prepared in C.2.2. Place the sample solution on a magnetic stirring platform and immerse the electrodes specified in C.2.2. Allow the sample solution to stir for a minimum of 15 seconds prior to taking a reading. Repeat the calibration check procedure using the 2 and 4 ppm fluoride standard solutions. Obtain the ppm F<sup>-</sup> ( $\mu$ g F<sup>-</sup>/mL) from the calibration curve.

C.2.2. <u>Calibration curve</u>. Prepare four standard solutions of fluoride ion ( $F^-$ ) by placing a 100 mL calibration line on four clean 4 oz. plastic screw cap bottles, using 100 mL of purified water. Discard over half of the water and quantitatively transfer 1.0, 2.0, 4.0, and 8.0 mL of the working sodium fluoride (NaF) standard, (100 µg  $F^-$ /mL), into the four marked bottles. Dilute to the mark with citrate buffer and mix. Using a specific ion meter equipped with fluoride specific ion and silver/silver chloride reference electrodes, obtain two consecutive reproducible readings (within ± 0.04 pH units) from each standard and plot meter reading versus micrograms of fluoride per milliliter ( $\mu g F^-$ /mL) using purified water as zero concentration.

C.2.3 Calculation. Use the following formula to calculate the HF content of the propellant sample:

$$HF, wt\% = \frac{0.264a}{W}$$

where,

a = Fluoride ion concentration in  $\mu$ g/mL.

W = Original propellant sample weight in (g) (4.3.3 & A.2).

#### C.2.4 Reagents and equipment.

C.2.4.1 <u>Reagents</u>. Commercially available analytical standards of suitable purity may be used where applicable.

a. Citrate buffer: Combine in beaker 57 mL ACS reagent grade glacial acetic acid, 58 g NaCl, and 0.3 g sodium citrate with 500 mL of purified 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 purified water.

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b. Stock standard F<sup>-</sup> solution: Dry several grams of ACS reagent grade sodium fluoride overnight in a vacuum oven at 100 °C (212 °F). Weigh out exactly 2.2105 g of dried NaF and dissolve it in purified water contained in 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<sup>-</sup> (1.0 mg F<sup>-</sup>/mL).

c. Working standard F<sup>-</sup> solution: Pipet 10.0 mL of solution b into a 100 mL volumetric flask and dilute to the mark with purified water. Transfer to a plastic screw cap bottle for storage. This solution contains 100 ppm F<sup>-</sup>(100  $\mu$ g F<sup>-</sup>/mL).

## C.2.4.2 Equipment.

- a. Electrodes, Ag/AgCl and F<sup>-</sup> ion specific.
- b. pH / ion-selective electrode (ISE) meter.

## MIL-PRF-7254H APPENDIX D

#### WATER

#### D.1 SCOPE

D.1.1 <u>Scope</u>. This Appendix describes a method for the determination of the water content in the propellant. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

## D.2 METHOD

D.2.1 <u>Procedure</u>. Obtain the absorption spectrum of the sample between 1500 and 1350 nm versus dichloromethane ( $CH_2CI_2$ ) both contained in the same cells used for the calibration. The water content is determined from the calibration curve prepared in accordance with D.2.2 and the calculation in D.2.3.

D.2.2 <u>Calibration curve</u>. Calibrate a double-beam spectrophotometer using 5 mm cells constructed in accordance with Figure D-1. Prepare a series of solutions of water in ACS reagent grade acetonitrile (CH<sub>3</sub>CN) at concentrations of 0.00, 0.01, 0.02, 0.03, 0.04, 0.06 g H<sub>2</sub>O/mL CH<sub>3</sub>CN and a series of reference beam compensating solutions substituting ACS reagent grade CH<sub>2</sub>Cl<sub>2</sub> for the water. Prepare the standards in accordance with Table D-I. Determine the absorbance of each H<sub>2</sub>O/CH<sub>3</sub>CN solution versus the matching CH<sub>2</sub>Cl<sub>2</sub>/CH<sub>3</sub>CN solution between 1500 and 1350 nm. Plot a curve through the points showing the absorbance ( $\Delta$ A) versus g H<sub>2</sub>O/mL CH<sub>3</sub>CN.

D.2.3 <u>Calculation</u>. Use the following formula to calculate the water content of the propellant:

$$H_2O, wt\% = \frac{D}{d} \times 100$$

Where:

D = Grams H<sub>2</sub>O/mL CH<sub>3</sub>CN from the calibration curve.

d = Relative density of the propellant (4.3.9).

g H <sub>2</sub> O/mL CH <sub>3</sub> CN solution	g H <sub>2</sub> O or mL CH <sub>2</sub> Cl <sub>2</sub>	mL CH₃CN solution
0.00	0.00	50.0
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

#### TABLE D-I. Standards

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FIGURE D-1. Stainless Steel Absorption Cell with Sapphire Windows

- D.2.4 Reagents and equipment.
- D.2.4.1 Reagents.
  - a. Acetonitrile, ACS reagent grade.
  - b. Dichloromethane, ACS reagent grade.

### D.2.4.2 Equipment.

- a. Spectrophotometer, double beam, equipped with 5 cm cells.
- b. Analytical balance calibrated to 0.01 g, 500 g capacity.

## MIL-PRF-7254H APPENDIX E

#### IRON

#### E.1 SCOPE

E.1.1 <u>Scope</u>. This Appendix describes a method for the determination of the iron content as  $Fe_2O_3$  in the propellant. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

## E.2 METHOD

E.2.1.2 <u>Procedure</u>. Add 5.0 mL of the propellant sample to a 25 mL volumetric flask containing approximately 15 mL of purified water. Mix the contents and dilute to the mark with purified water and mix again. Determine the percent absorption of the sample using a suitable atomic absorption spectrophotometer which has been set at an iron wavelength closest to 2480 Å.

E.2.1.1 <u>Calibration</u>. Construct a calibration curve by preparing a 5, 10 and 20  $\mu$ g/mL (ppmv) Fe standard in a 5 N HNO<sub>3</sub> solution and store in plastic screw-cap bottles. After setting the instrument to zero absorbance with 5 N HNO<sub>3</sub>, determine the absorbance of the calibration standards ( $\lambda \approx 2480$ Å). Prepare a calibration curve of absorbance vs iron concentration.

E.2.1.3 <u>Calculation</u>. The iron oxide ( $Fe_2O_3$ ) content of the propellant sample shall be calculated as follows:

$$Fe_2O_3 wt\% = \frac{ppmv Fe(1.43)(V_d)}{(V_h)(d)(1 \times 10^4)}$$

Where:

 $V_d$  = Final dilution volume, mL.

 $V_h$  = Volume of the propellant sample used, mL.

d = Relative density of the propellant (4.3.9).

E.2.1.4 <u>Reagents and equipment</u>. The following shall apply as test conditions of E.1.

E.2.1.4.1 <u>Reagents.</u> Commercially available analytical standards of suitable purity may be used where applicable.

a. Hydrochloric acid, 6 N. Add 50 mL ACS reagent grade 37% (average) concentrated HCI to 50 mL purified water; store in a plastic bottle.

b. Nitric acid, 5 N. Dilute 320 mL of concentrated (70%) ACS reagent grade  $HNO_3$  in a 1 L volumetric flask containing 500 mL of purified water, mix the solution thoroughly and dilute to the mark with purified water. Mix again and pour into a plastic bottle for storing.

c. Iron standard solution, 1000  $\mu$ g/mL. Weigh 1.000 g of 99.99% purity iron wire or shot. Transfer to a 1 L volumetric flask, add 17 mL of 6 N HCl solution, heat on a hotplate until the iron dissolves and dilute to the mark with 5 N HNO<sub>3</sub>.

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## E.2.1.4.2 Equipment.

- a. Hotplate/stirrer controllable heat and stir settings.
- b. Analytical balance calibrated to 0.1 mg.
- c. Atomic absorption spectrophotometer with an iron hollow cathode lamp.

## MIL-PRF-7254H APPENDIX F

#### SOLIDS

#### F.1 SCOPE

F.1.1 <u>Scope</u>. This Appendix describes a method for the determination of the solids content in the propellant. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

## F.2 METHOD

F.2.1.1 <u>Procedure</u>. Mix the propellant sample thoroughly and immediately transfer exactly 60.0 mL or two 30.0 mL aliquots to a clean platinum crucible which has been accurately tared to the nearest 0.1 mg. Add 1 mL of concentrated  $H_2SO_4$  and slowly evaporate the acid on a hot plate in a fume hood until dense fumes of sulfur trioxide evolve. Transfer the crucible from the hot plate to a triangle over a Meeker burner to continue the decomposition of the sulfates before ignition. Place the residue in a muffle furnace at 900 °C (1652 °F) for 30 minutes to a constant weight. Alternatively, a Meeker burner may be used.

F.2.1.2 <u>Calculation</u>. The solids shall be calculated by the following formula:

Solids, 
$$wt\% = \frac{g \times 100 \times a}{60 \times d}$$

Where:

g = weight of residue in grams.

a = 4.18 for aluminum and 3.03 steel containers.

d = Relative density of the propellant (4.3.9).

F.2.1.3 <u>Reagents and equipment</u>. The following shall apply as test conditions of F.1.

#### F.2.1.3.1 Reagents.

a. Sulfuric acid, 98% ACS reagent grade.

#### F.2.1.3.2 Equipment.

- a. Platinum crucible, 50 100 mL capacity.
- b. Hotplate.
- c. Triangle, silica.
- d. Tripod.
- e. Muffle furnace, Meeker burner or equivalent.
- f. Analytical balance accurate to 0.1 mg, 200 g capacity.

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## MIL-PRF-7254H

CONCLUDING MATERIAL

Custodians: Army – MI Navy – AS Air Force – 68 DLA – PS Preparing Activity Air Force – 68 (Project: 9135-2014-001)

Review Activities Air Force – 11, 19 Navy – SA

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 <a href="https://assist.dla.mil">https://assist.dla.mil</a>.