

NOT MEASUREMENT SENSITIVE

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PERFORMANCE SPECIFICATION  
PROPELLANT, HYDROGEN PEROXIDE

1. SCOPE.

1.1 Scope. This specification covers four types and two grades of hydrogen peroxide propellant.

1.2 Classification. Hydrogen peroxide propellant will be of the following designated types and grades:

Types

70 – 71.0 to 73.0 percent hydrogen peroxide  
85 – 85.0 to 87.0 percent hydrogen peroxide  
90 – 89.5 to 91.0 percent hydrogen peroxide  
98 – 98.0 to 99.0 percent hydrogen peroxide

Grades

ES – Extra Stabilizers: contains higher concentration of stabilizers  
HP – High Purity: stricter limits on type and concentration of stabilizers/metals

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in Sections 3 and 4 of this specification. This section does not include documents 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 documents cited in sections 3 and 4 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Code PS, DESC-BPE RM 2954, Defense Energy Support Center, 8725 John J Kingman Road, Fort Belvoir VA 22060-6222, by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

FSC 9135

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those listed in the issue of the Department of Defense Index of Specifications and Standards (DoDISS) and supplement thereto, cited in the solicitation (see 6.2)

## SPECIFICATIONS

### DEPARTMENT OF DEFENSE

#### MIL-PRF-27401 – Propellant, Pressurizing Agent, Nitrogen

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 the documents which are DoD adopted are those listed in the issue of the DoDISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS are the issues of the documents cited in the solicitation (see 6.2).

### AMERICAN CHEMICAL SOCIETY (ACS)

#### ACS Reagent Chemical Standards

(Application for copies should be addressed to the American Chemical Society, 1 River Rd, Niskayuna NY 12309.)

### AMERICAN SOCIETY FOR QUALITY CONTROL

#### ANSI/ASQC Z1.4 – Sampling Procedures and Tables for Inspection by Attributes

(Application for copies should be addressed to the American Society for Quality Control, 611 East Wisconsin Avenue, Milwaukee WI 53202.)

### AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

- ASTM D 1193 - Standard Specification for Reagent Water
- ASTM D 2579 - Standard Test Method for Total Organic Carbon in Water
- ASTM D 3919 - Standard Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry
- ASTM D 5542 - Standard Test Method for Trace Anions in High Purity Water by Ion Chromatography
- ASTM D 5673 - Standard Test Method for Elements in Water by Inductively Coupled Plasma – Mass Spectrometry
- ASTM E 29 - Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

(Application for copies should be addressed to the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken PA 19428-2959.)

### INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

- ISO 14911 - Water Quality — Determination of Dissolved  $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{NH}_4^+$ ,  $\text{K}^+$ ,  $\text{Mn}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Sr}^{2+}$  and  $\text{Ba}^{2+}$  Using Ion Chromatography

(Application for copies should be addressed to the International Organization for Standardization, Case Postale 56 · CH-1211, Genève 20 · Switzerland, or Email to: iso@iso.ch)

2.4 Order of precedence. 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.

TABLE I. Chemical and physical properties.

| Properties   | Limits      |             |             |             |             |
|--|-------------|-------------|-------------|-------------|-------------|
|  | Type 70     | Type 85     | Type 90     |             | Type 98     |
|  | Grade ES    | Grade ES    | Grade ES    | Grade HP    | Grade HP    |
| Hydrogen peroxide assay<br>(percent by weight)             | 71.0 - 73.0 | 85.0 - 87.0 | 90.0 - 91.5 | 90.0 - 91.5 | 98.0 - 99.0 |
| <b>Anions</b>  |             |             |             |             |             |
| Chloride (Cl <sup>-</sup> ) mg/kg                          | 0.8 max     | 0.2 max     | 2 max       | 0.5 max     | 0.5 max     |
| Nitrate (NO <sub>3</sub> <sup>-</sup> ) mg/kg <sup>1</sup> | 3.9 max     | 5.0 max     | 7.5 max     | 5.0 max     | 5.0 max     |
| Phosphate (PO <sub>4</sub> <sup>-3</sup> ) mg/kg           | 0.2 max     | 0.2 max     | 0.5 max     | 0.2 max     | 0.2 max     |
| Sulfate (SO <sub>4</sub> <sup>-2</sup> ) mg/kg             | 2.3 max     | 0.5 max     | 5 max       | 0.5 max     | 0.5 max     |
| Ammonium (NH <sub>4</sub> <sup>+</sup> ) mg/kg             | 2.3 max     | 3.0 max     | 3.0 max     | 3.0 max     | 3.0 max     |
| Stability (24 h/100°C)<br>% loss of active oxygen          | 2 max       | 2 max       | 2 max       | 2 max       | 2 max       |
| Evaporation residue mg/kg                                  | 15 max      | 10 max      | -           | 20 max      | 20 max      |
| Total Carbon, mg/l   | 200 max     | 40 max      | 105 max     | 40 max      | 40 max      |
| <b>Metals:</b>   |             |             |             |             |             |
| Aluminum (Al) mg/l   | 0.2 max     | 0.2 max     | 1.0 max     | 0.35 max    | 0.35 max    |
| Tin (Sn) mg/l  | 0.8 - 3.1   | 1.0 - 4.0   | 0.7 - 7.0   | 1.0 - 4.0   | 1.0 - 4.0   |
| Chromium (Cr) mg/l   | 0.11 max    | 0.03 max    | -           | 0.03 max    | 0.03 max    |
| Lead (Pb) mg/l   | 0.04 max    | 0.03 max    | -           | 0.03 max    | 0.03 max    |
| Manganese (Mn) mg/l  | 0.04 max    | 0.03 max    | -           | 0.03 max    | 0.03 max    |
| Iron (Fe) mg/l   | 0.11 max    | 0.03 max    | -           | 0.03 max    | 0.03 max    |
| Copper (Cu) mg/l   | 0.04 max    | 0.03 max    | -           | 0.03 max    | 0.03 max    |
| Nickel (Ni) mg/l   | 0.04 max    | 0.03 max    | -           | 0.03 max    | 0.03 max    |
| Antimony (Sb) mg/l   | -           | -           | -           | 0.03 max    | 0.03 max    |
| Arsenic (As) mg/l  | -           | -           | -           | 0.03 max    | 0.03 max    |
| Gold (Au) mg/l   | -           | -           | -           | 0.03 max    | 0.03 max    |
| Zinc (Zn) mg/l   | -           | -           | -           | 0.03 max    | 0.03 max    |
| Titanium (Ti) mg/l   | -           | -           | -           | 0.03 max    | 0.03 max    |

<sup>1</sup> High purity grade shall be available with a minimum level of 2.0 mg/kg nitrates when requested.

### 3. REQUIREMENTS

3.1 Chemical composition and physical properties. The chemical composition and physical properties of the propellants shall conform to Table I when tested in accordance with the applicable test methods.

3.2 Additives. For the high purity grades of the propellant, the only additives permitted as stabilizers are reagent grade (>99%) sodium nitrate (NaNO<sub>3</sub>), reagent grade (>98%) potassium stannate trihydrate (K<sub>2</sub>SnO<sub>3</sub> · 3H<sub>2</sub>O) and reagent grade (>98%) sodium stannate trihydrate (Na<sub>2</sub>SnO<sub>3</sub> · 3H<sub>2</sub>O).

3.3 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 E 29 for Using Significant Digits in Test Data to Determine Conformance with Specifications.

3.4 Qualitative. The propellant shall be a clear, colorless, homogenous liquid when examined visually by transmitted light.

#### 4. VERIFICATION

4.1 Classification of inspections. The inspection requirements specified herein are classified as quality conformance inspections. (see 4.2)

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

- a. Examination ..... 4.2.1
- b. Laboratory Analysis ..... 4.2.2

4.2.1 Examination. The propellant shall be subject to the following test as described under 4.4.

Examination of product ..... 4.4.1

4.2.2 Laboratory Analysis. The propellant shall be sampled according to 4.2.2.1 and the samples tested for conformance to the limits of Table I utilizing the procedures described under 4.4.

4.2.2.1 Sampling plan.

4.2.2.1.1 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 which 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 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.

4.2.2.1.2 Sample. Three one-liter samples of propellant shall be selected as specified in 4.2.2.1.3 or 4.2.2.1.4. Each sample taken shall be placed in a clean, clear, glass container, examined visually and tested for hydrogen peroxide assay. The sample with the lowest assay shall be subjected to the additional tests of Table I. Quality conformance tests shall be made upon the samples of propellant taken directly from shipping containers at the place of manufacture and time of shipment.

4.2.2.1.3 Drums. Samples shall be drawn by means of an accepted sampling device. The number of drums selected from each lot shall be according to ANSI/ASQC Z1.4, inspection level S-3. The sample shall be taken approximately one inch below the surface of the liquid. If more than one lot is represented in a shipment, then each lot represented shall be considered as a separate lot for sampling purposes. The contents of each selected drum shall be thoroughly mixed by agitation in an upright position immediately prior to sampling.

4.2.2.1.4 Tanks. Samples shall be taken by means of a sampling thief or other accepted device from each portable tank, cargo tank, or tank car. One sample shall be drawn within one foot of the surface, one near the midpoint, and one within one foot of the bottom.

4.3 Rejection. When any sample of the propellant tested in accordance with 4.2 fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected.

#### 4.4 Test methods.

4.4.1 Examination of Product. One twenty-five milliliter (ml) specimen shall be taken from each of the samples and shall be placed in separate laboratory glass test tubes. Each separate test tube shall be visually inspected across the diameter of the tube and compared with a similar tube of reagent water (ASTM D 1193 Type 1) to verify conformance with this specification as to clarity and color. (see 3.4).

4.4.2 Hydrogen peroxide assay. Hydrogen peroxide assay shall be determined by one of the following procedures. In the event of dispute, the procedure specified in paragraph 4.4.2.1 shall be the referee method.

##### 4.4.2.1 Assay by titration.

4.4.2.1.1 Procedure. Accurately weigh about 1 ml of sample in a tared 100ml volumetric flask, dilute to volume with reagent water (ASTM D 1193 Type 1) and mix thoroughly. Transfer exactly 20.0 ml of this solution to a 250 ml conical flask, add 20 ml of 2.5N H<sub>2</sub>SO<sub>4</sub> solution and titrate with standardized 0.5 N (0.1M) potassium permanganate to a pink color that persists for 15 seconds. Correct the volume by titrating a blank aliquot to the same endpoint. The titrant shall be at the same temperature during the assay and the standardization.

4.4.2.1.2 Calculation. The percent H<sub>2</sub>O<sub>2</sub> by weight shall be calculated by the following formula:

$$\%H_2O_2 = \frac{8.5 \cdot V_{KMnO_4} \cdot N_{KMnO_4}}{W_{H_2O_2}}$$

where

$V_{KMnO_4}$  = corrected volume(ml) of potassium permanganate

$N_{KMnO_4}$  = normality of potassium permanganate

$W_{H_2O_2}$  = weight of sample (g)

4.4.2.1.3 Standardization of potassium permanganate solution. Bring 500 ml of dilute sulfuric acid (one volume concentrated acid plus 19 volumes of reagent water) to a boil and allow to cool. Separate into two 250ml aliquots. Dissolve about 1.50 g, weighed to the nearest 0.1 mg, of reagent grade sodium oxalate (dried at 105°C) into one of the aliquots and save the other aliquot as a blank. From a burette, add 39 - 40 ml of the potassium permanganate solution to the oxalate solution at a rate of 25 – 35 ml per minute while stirring slowly. Allow the solution to stand until the pink color has disappeared, then heat to 55-60°C and continue the titration, while stirring slowly, until a faint pink color (sighted on a white background) persists for 30 seconds. Correct the volume by titrating the blank acid aliquot to the same endpoint. Calculate the normality of the potassium permanganate solution as follows:

$$N_{\text{KMnO}_4} = \frac{14.92 \cdot W_{\text{C}_2\text{Na}_2\text{O}_4}}{V_{\text{KMnO}_4}}$$

where

$$\begin{aligned} N_{\text{KMnO}_4} &= \text{Normality of potassium permanganate solution} \\ W_{\text{C}_2\text{Na}_2\text{O}_4} &= \text{Weight of sodium oxalate (g)} \\ V_{\text{KMnO}_4} &= \text{Corrected titrated volume of potassium permanganate solution (ml)} \end{aligned}$$

4.4.2.2 Assay by densitometer. The procuring activity may accept determination of hydrogen peroxide assay by density measurement provided a statistical comparison of assay by density versus assay by titration has been performed and the appropriate conversion tables/equations have been developed and provided to the procuring activity. The comparison must show that the assay can be predicted reliably within  $\pm 0.1\%$ . A separate density to assay conversion calculation must be performed for each type and grade of product. Density measurements must be made within an accuracy of  $\pm 0.00002$  g/ml. Any changes to the manufacturing process for each type and grade of product will require a new statistical comparison of density versus assay by titration.

4.4.3 Sample decomposition. The test methods for anions and ammonium require the concentrated hydrogen peroxide to be decomposed prior to the analysis. Decompose the sample utilizing either of the following procedures.

4.4.3.1 Evaporation. Accurately weigh about 40 ml of the hydrogen peroxide sample in a porcelain or glass evaporation dish. Cover the dish with a ribbed watch glass and decompose the hydrogen peroxide by carefully evaporating over a steam bath. Rinse the sides of the dish and the watch glass with reagent water (ASTM D 1193 Type 1) and again evaporate to dryness. Add 10 ml of reagent water and heat on the steam bath to dissolve the residue. Quantitatively transfer the contents of the dish to a 50-ml volumetric flask. Rinse the dish and the watch glass at least twice and transfer the washings to the flask. Dilute to the mark with reagent water and label as decomposed solution. Prepare a blank solution by treating a 40 ml volume of reagent water in an identical manner.

4.4.3.1.1 Calculation. The concentration of hydrogen peroxide in the decomposed solution shall be calculated using the following formula:

$$C_{\text{H}_2\text{O}_2} = \frac{W_{\text{H}_2\text{O}_2}}{V}$$

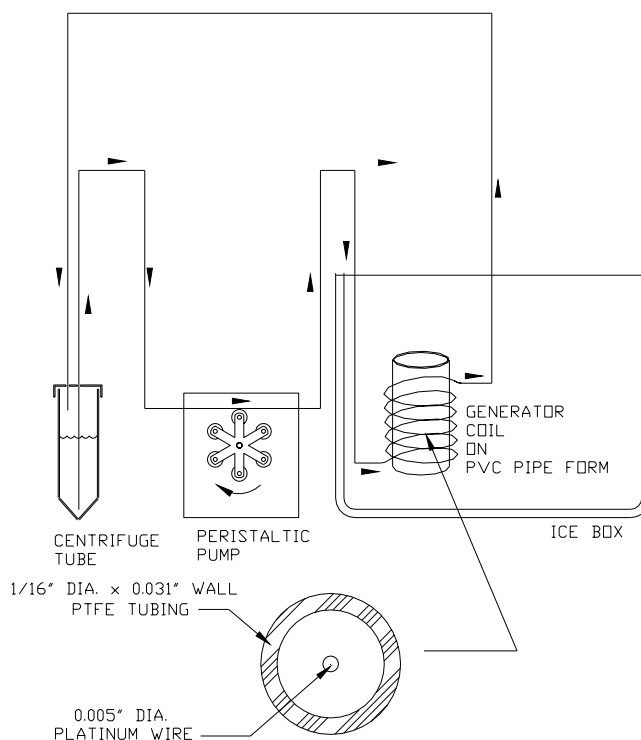
where

$$\begin{aligned} C_{\text{H}_2\text{O}_2} &= \text{Concentration of hydrogen peroxide in decomposed solution (g/ml)} \\ W_{\text{H}_2\text{O}_2} &= \text{Weight of sample (g)} \\ V &= \text{Volume of decomposed solution (ml)} \end{aligned}$$

4.4.3.2 Platinum wire catalyzed decomposition in PTFE tubing. This method allows unassisted decomposition of the concentrated hydrogen peroxide utilizing the apparatus described below.

4.4.3.2.1 Assembly of Apparatus. Obtain a ~20 foot length of 0.125 inch OD, 0.063 inch ID PTFE tubing. Push a length of 0.032 inch OD stainless steel safety wire through the PTFE tubing. It is helpful to extend the PTFE tubing out straight. When the end of the stainless steel safety wire comes out the end of the PTFE tubing, use a cyanoacrylate adhesive (like Super Glue) to attach the end of a 16 foot length of 0.005 inch OD platinum wire to the safety wire. Allow the glue to set well. Gently pull the safety wire back through the PTFE tube until the platinum wire is centered in the PTFE tubing. Squirt some

acetone into the end of the PTFE tubing where the safety wire is protruding and allow it to flow down to the adhesive. Allow the acetone to attack the adhesive for a few minutes or until the bond is broken, then pull the safety wire out, leaving the platinum wire in the PTFE tubing. You should have a ~2 foot pigtail of PTFE tubing with no platinum wire in it at both ends of the 20 foot length now. Flow deionized water through the tubing to remove the acetone, then dry the tubing with a gaseous nitrogen stream. Obtain a ~5 inch length of 3 inch ID PVC pipe, and starting near one end, coil the platinum filled PTFE tubing around it. Leave the pigtails free from the coil. Secure the coil at both ends of the PVC pipe with a loop of safety wire so it does not unwind or change position. Obtain a 50 ml centrifuge tube and put 2 holes through the lid that are large enough to clear the PTFE tubing. Obtain a peristaltic pump that has a flow range from about 0.5 ml per minute to at least 15 ml per minute and an ice chest about 12 x 8 x 8 inch internal dimensions. Set up the apparatus as shown in Figure 1.



**FIGURE 1. Decomposition Assembly**

**4.4.3.2.2 Preparation.** Temporarily remove the inlet and outlet ends of the coil from the centrifuge tube. Place the inlet end in a beaker of with at least 500 ml of 5% nitric acid in it, place the outlet end in a suitably sized waste beaker then pump the 5% nitric acid through the coil. Use a pump speed of greater than 15 ml per minute. Repeat with 1000 ml of deionized water. Put a gentle stream of  $\text{GN}_2$  through the tubing to dry it. Reassemble the apparatus as shown in Figure 1.

**4.4.3.2.3 Procedure.**

**4.4.3.2.3.1 Preparing the Sample.** Weigh the centrifuge tube to the nearest 0.01 grams and record this as the tare weight. Transfer a volume of hydrogen peroxide to the centrifuge tube that should be close to 50 grams, if the concentration is known and no more than 40 ml in any case. 70% hydrogen peroxide corresponds to about 40 ml, 90% is about 36 ml and 100% would be about 35 ml. Weigh the

centrifuge tube again, and record this as the gross weight of the sample. Place the centrifuge tube in a heavy walled 250 ml beaker to keep it upright, and the beaker in a plastic tub for secondary containment. Reassemble the apparatus as shown in Figure 1. Using Calpico™ tape (or equivalent), secure the return side of the PTFE tubing in the ullage of the centrifuge tube. If this tubing should come out, hydrogen peroxide would be released into the secondary container. Ensure that the suction side of the tubing is pushed to the bottom of the centrifuge tube. Fill the ice box with ice then add water to it so that it is a slush, and ensure that the coil is oriented so that the inlet is at the bottom of the PVC form.

4.4.3.2.3.2 Decomposing the Sample. Turn on the peristaltic pump with a flow of about 0.5 ml per minute. Observe the system until gas and liquid are seen returning to the centrifuge tube. This should only be a matter of minutes from when the hydrogen peroxide reaches the coil. Allow the system to run until no more bubbles are observed in the effluent stream. Typically, this will be accomplished if the system runs overnight. An 8-hour work shift is not enough time.

4.4.3.2.3.3 Flushing the Apparatus. After the hydrogen peroxide has decomposed, remove the suction end of the PTFE tubing from the centrifuge tube, and then increase the pump speed to about 10 ml per minute. Lift the coil out of the ice box and invert it so that the outlet of the coil is near the bottom of the PVC form. Elevate the coil above the top of the centrifuge tube so that gravity assists the draining process. Once the water has drained out of the coil, check the volume of water left after the reaction, place 10 to 15 ml (make sure you do not exceed the capacity of the centrifuge tube) of reagent water (ASTM D 1193 Type 1) in a clean beaker, and allow it to be pumped through the system. Allow the reagent water to drain into the centrifuge tube. Shut off the peristaltic pump, remove the tubing, and then apply a gentle gaseous nitrogen purge to the tubing to push out the last of the water. When the water droplets stop coming out of the tubing, remove the nitrogen flow from the coil. Adjust the volume in the centrifuge tube to the 50 ml mark with reagent water and label as decomposed solution.

4.4.3.2.3.4 Preparing the Blank Controls. Reconnect the tubing to the peristaltic pump, the coil may be left inverted, and pump 50 ml of reagent water through the system into a centrifuge tube labeled as a system blank. Fill a third centrifuge tube with the reagent water and label as a reagent blank.

4.4.3.2.4 Calculation. The concentration of hydrogen peroxide in the decomposed solution shall be calculated by the following formula:

$$C_{H_2O_2} = \frac{G - T}{V}$$

where

$C_{H_2O_2}$  = Concentration of hydrogen peroxide in decomposed solution (g/ml)

$G$  = Gross weight - centrifuge tube with sample (g)

$T$  = Tare weight of centrifuge tube (g)

$V$  = Volume of decomposed solution (ml)

4.4.4 Anions. This test method covers the determination of trace levels of chloride, nitrate, phosphate and sulfate in hydrogen peroxide by ion chromatography. The concentration of these anions is determined by analyzing the decomposed solution prepared in 4.4.3 in accordance with ASTM D 5542. Calculate the concentration of each anion in the hydrogen peroxide sample using the following formula:

$$A^- = \frac{R}{1000 \cdot C_{H_2O_2}}$$

where



- $A^-$  = Concentration of anion in the hydrogen peroxide sample (mg/kg)  
 $R$  = Result from ASTM procedure for anion concentration ( $\mu\text{g/l}$ )  
 $C_{H_2O_2}$  = Concentration of hydrogen peroxide in decomposed solution (g/ml)

4.4.5 Ammonium. Ammonium concentration is determined by analyzing the decomposed solution, prepared in 4.4.3, in accordance with ISO 14911 with the exception that only the concentration of the ammonium cation needs to be determined. Calculate the ammonium concentration in the hydrogen peroxide sample using the following formula:

$$NH_4^+ = \frac{R}{C_{H_2O_2}}$$

where

- $NH_4^+$  = Concentration of ammonium in the hydrogen peroxide sample (mg/kg)  
 $R$  = Result from ISO procedure for ammonium concentration (mg/l)  
 $C_{H_2O_2}$  = Concentration of hydrogen peroxide in decomposed solution (g/ml)

#### 4.4.6 Stability.

4.4.6.1 Procedure. Transfer 50 ml of the sample into each of three 50-ml Pyrex volumetric flasks that have been specifically prepared according to 4.4.6.3. Cover the top of each flask immediately with a 5 to 10-ml Pyrex beaker across its mouth to prevent condensed droplets from escaping. Weigh the flask accurately to 0.01 gram. Place the flasks in a boiling water or steam bath. The flask necks must be sealed to prevent the steam from heating the upper section so that sufficient condensing surface is available to the samples. After 24 hours at the specified temperature remove the flasks. Cool to ambient temperature and reweigh to  $\pm 0.01$  gram.

4.4.6.2 Calculation. The percent loss of active oxygen is calculated by averaging the results of the individual flasks using the following formula:

$$\Delta\%O_2 = \frac{100(W_1 - W_2)}{0.470CW_1}$$

where

- $\Delta\%O_2$  = percent active oxygen loss  
 $W_1$  = initial net weight  
 $W_2$  = final net weight  
 $C$  = weight fraction of sample (concentration/100)

4.4.6.3 Test flask preparation. Dry the test flask and cover it with a 5 to 10-ml pyrex beaker. Place the covered flask on a balance and record the tare weight to the nearest 0.01 gram. Fill the flask with concentrated nitric acid, cover the top of the flask with aluminum foil, and place an inverted small beaker over the aluminum cover. Place the flask in a boiling water or steam bath. Pour off the nitric acid and rinse the flask with distilled water immediately followed with a hydrogen peroxide rinse. Distilled water shall not be allowed to dry in the flask. Screen the flask by performing the stability test using

samples. Flasks that give inconsistent results shall be rejected. Flasks that give consistent results shall only be used for hydrogen peroxide samples.

#### 4.4.6.4 Reagents and equipment.

##### a. Reagents:

Nitric acid: Concentrated, ACS reagent grade.

##### b. Equipment:

- (1) Flask: Volumetric, pyrex, 50-ml, 3 required with 4-inch extension on flask neck.
- (2) Aluminum: Foil, pure.
- (3) Balance: Analytical, 0.1 mg sensitivity.
- (4) Water or steam bath.
- (5) Beaker: Glass, small, 3 required.

#### 4.4.7 Evaporation residue.

4.4.7.1 Procedure. Add several pieces of clean platinum that have a surface area equivalent to the inside of two 100-ml casseroles into a clean 2-liter pyrex beaker containing 500 ml of reagent water (ASTM D 1193 Type 1). Cover the beaker with a clean, ribbed watch glass and place it on a hot plate. Add 300 ml of the sample in not larger than 50-ml increments. Between increments, allow the major portion of each increment to decompose. If the solution is maintained at boiling, decomposition of the total solution will require 2 to 4 hours. Immediately prior to total decomposition, transfer the solution to a clean 1-liter Pyrex boiling flask and cover it with a clean ribbed watch glass. Add a small piece of clean platinum to the solution to prevent concentration of any remaining hydrogen peroxide in the sample. Boil the solution until condensed to a 25 to 50-ml volume. Transfer the remaining solution to a 100-ml platinum evaporating dish that has attained constant weight by heating. Allow the solution to completely evaporate. While decomposing and condensing the sample, add 500 ml of reagent water to a clean 1-liter Pyrex boiling flask and cover it with a clean ribbed watch glass. Place the flask on a hot plate and boil until condensed to a 25 to 50-ml volume. Discontinue boiling and transfer the remaining water to a 100-ml platinum dish that has attained constant weight by heating. Place the dishes in a vacuum oven at 105° to 110°C (221° -230°F) for a minimum of 1 hour. Remove the dishes and allow them to cool in a desiccator. Place on a balance and record the weight to the nearest 0.1 milligram.

4.4.7.2 Calculation. The evaporation residue of the sample shall be calculated as follows:

$$ER = \frac{W_R}{0.3}$$

where

$ER = \text{residue in mg/L}$

$W_R = \text{corrected weight of residue in mg}$

#### 4.4.7.3 Reagents and equipment.

##### a. Reagents:

Water: ASTM D 1193 Type 1

## b. Equipment:

- (1) Beaker, Pyrex, 2-liter capacity
- (2) Watch glass, ribbed, 3 required
- (3) Hot plate
- (4) Flask, boiling, Pyrex, 1-liter capacity, 2 required.
- (5) Dish, evaporating, platinum, 100-ml capacity, 2 required
- (6) Oven, vacuum
- (7) Desiccator containing calcium sulfate
- (8) Balance, analytical, 0.1 mg sensitivity

4.4.8 Total Carbon. Determine the total carbon by following the procedures outlined in ASTM D 2579 with the exception that a neat sample of hydrogen peroxide shall be used instead of water.

4.4.9 Metals. Concentration of metals in the sample shall be determined by one of the following procedures. In the event of dispute, the procedure specified in paragraph 4.4.9.1 shall be the referee method.

4.4.9.1 Inductively Coupled Plasma. Determine concentration of metals by inductively coupled plasma-mass spectrometry following the procedures outlined in ASTM D 5673 with the exception that a neat sample of hydrogen peroxide shall be used instead of water.

4.4.9.2 Atomic Absorption. Determine concentration of metals by graphite furnace atomic absorption spectrophotometry following the procedure outlined in ASTM D 3919 with the exception that a neat sample of hydrogen peroxide shall be used instead of water.

## 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 personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. 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 specification is intended for use as an oxidizer or monopropellant in rocket engines.

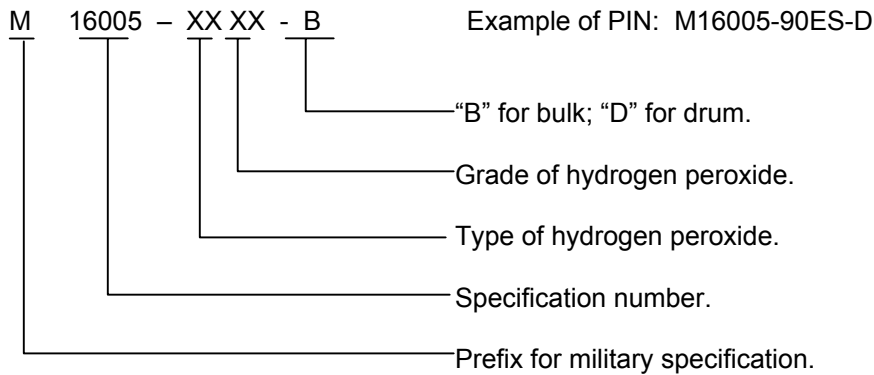
6.2 Acquisition requirements. Acquisition documents must specify the following:

- a. Title, number, and date of this specification.
- b. Type of propellant.
- c. Grade of propellant if applicable.

- d. Issue of DoDISS to be cited in the solicitation, and, if required, the specific issue of individual documents referenced (2.2.1 and 2.3).
- e. Approved method for determination of hydrogen peroxide assay (4.4.2).
- f. Method of shipment, type and capacity of containers.
- g. Quantity by weight.
- h. Packaging requirements (see 5.1).

6.3 Part Identification Number (PIN). The following part identification numbering procedure is for government purposes and does not constitute a requirement for the contractor.

This example describes a part numbering system.



CONCLUDING MATERIAL

Custodians  
 Navy – AS  
 Air Force – 68  
 Army – MI

Review Activities  
 Air Force – 11, 99  
 Navy – SA

Preparing Activity  
 DLA – PS

Civil Agency Interest  
 GSA – 6FEE  
 NASA – NA

(Project 9135-0163)

## STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

### INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.
2. The submitter of this form must complete blocks 4, 5, 6, and 7, and send to preparing activity.
3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements.

|  |   |  |
|--|---|--|
| <b>I RECOMMEND A CHANGE:</b>   | 1. DOCUMENT NUMBER<br>MIL-PRF-16005F  | 2. DOCUMENT DATE (YYYYMMDD)            |
| 3. DOCUMENT TITLE<br>PROPELLANT, HYDROGEN PEROXIDE   |   |  |
| 4. NATURE OF CHANGE <i>(Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)</i> |   |  |
| 5. REASON FOR RECOMMENDATION   |   |  |
| 6. SUBMITTER   |   |  |
| a. NAME <i>(Last, First, Middle Initial)</i>   | b. ORGANIZATION   |  |
| c. ADDRESS <i>(Include ZIP Code)</i>   | d. TELEPHONE <i>(Include Area Code)</i><br>(1) Commercial<br>(2) DSN<br><i>(If applicable)</i>  | 7. DATE SUBMITTED<br><i>(YYYYMMDD)</i> |
| 8. PREPARING ACTIVITY  |   |  |
| a. NAME<br>DESC-BPE  | b. TELEPHONE <i>(Include Area Code)</i><br>(1) Commercial<br>(2) DSN  |  |
| c. ADDRESS <i>(Include ZIP Code)</i>   | <b>IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT:</b><br>Defense Standardization Program Office (DLSC-LM)<br>8725 John J. Kingman Road, Suite 2533<br>Fort Belvoir, Virginia 22060-6221<br>Telephone (703) 767-6888      DSN 427-6888 |  |