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MILITARY SPECIFICATION
PROPELLANT, HYDROGEN PEROXIDE

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

1. SCOPE

1.1 Scope. This specification covers hydrogen peroxide (H_2O_2)
propellant.

1.2 Classification. Hydrogen peroxide covered by this specifi-
cation shall be of the following types:

Hydrogen peroxide assay (percent by weight)

Type I - 91.0 max, 90.0 min

Type II - 99.0 max, 98.0 min

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issue in effect on date of
invitation for bids or request for proposal, form a part of this speci-
fication to the extent specified herein.

SPECIFICATIONS

Military

MIL-P-25508

Propellant, Oxygen

Federal

RR-S-366

Sieves, Standard for Testing Purposes

STANDARDS

Military

MIL-STD-105

Sampling Procedures and Tables for
Inspection by Attributes

MIL-STD-129

Marking for Shipment and Storage

MIL-STD-172

Color Code for Containers of Liquid Propellants

(Copies of documents required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issues in effect on date of invitation for bids or request for proposal shall apply.

Department of Transportation

49 CFR 171-190

Department of Transportation
Hazardous Materials Regulations

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

American Society for Testing and Materials Publications

ASTM Standards, Parts 8, 17, and 30.

(Copies of ASTM Publications may be obtained upon application to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

3. REQUIREMENTS

3.1 Chemical properties. The chemical properties of the propellant shall conform to those listed in table I when tested in accordance with the applicable test methods.

3.2 Drum inspection. Drums containing propellant shall be free from defects, leakage, and any indications of propellant decomposition when inspected in accordance with 4.1.1.

3.3 Limiting values. For purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limitation value, in accordance with the rounding-off method of the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E29).

TABLE I
Physical and Chemical Requirements

Properties	Limits Type I	Limits Type II	Test Paragraphs
Hydrogen Peroxide assay (% by weight)	91.0 max 90.0 min	99.0 max 98.0 min	4.5.2
Aluminum (Al) (Milligram per liter) mg/l	0.5 max	0.5 max	4.5.4
Chloride (Cl ⁻) mg/l	1.0 max	1.0 max	4.5.5
Ammonium (NH ₄ ⁺) mg/l	3.0 max	3.0 max	4.5.6
Nitrate (NO ₃ ⁻) mg/l	5.0 max 3.0 min	5.0 max 3.0 min	4.5.7
Phosphate (PO ₄ ⁻³) mg/l	0.2 max	0.2 max	4.5.8
Sulfate (SO ₄ ⁻²) mg/l	3.0 max	3.0 max	4.5.9
Tin (Sn) mg/l	4.0 max 1.0 min	4.0 max 1.0 min	4.5.10
Carbon (C) mg/l	200 max	200 max	4.5.11
Evaporation residue mg/l	20 max	20 max	4.5.12
Stability	2.0% max of active O ₂	2.0% max of active O ₂	4.5.13
Particulate mg/l	1.0 max	1.0 max	4.5.14

3.4 Filter. A filter with a 10-micron nominal and 40 absolute rating shall be installed between the manufacturer's plant system and the container to be filled for delivery.

3.5 Qualitative. The propellant shall be a colorless liquid when examined visually by transmitted light.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.1.1 Inspection of filled drums. Each filled drum selected in accordance with 4.4.2.1.3 shall be examined for defects of the drum and the closure for evidence of leakage, and for unsatisfactory markings; each filled drum shall also be weighed to determine the amount of the contents. The tare weights furnished by the manufacturer shall be accepted and tare weights shall not be verified unless the Government inspector has evidence that these may not be correct. Each selected drum shall be inspected to detect any unusual temperature or sound of activity caused by the propellant; these are signs of decomposition of the propellant, and any drum showing either shall be rejected.

4.2 Classification of tests. The inspection and testing of the propellant shall be classified as quality conformance tests.

4.3 Test conditions. The test conditions are described under the individual tests to which they apply.

4.4 Quality conformance tests. Quality conformance tests shall consist of:

(a) Individual test 4.4.1

(b) Sampling test 4.4.2

4.4.1 Individual test. The propellant shall be subject to the following test as described under 4.5.

Examination of product 4.5.1

4.4.2 Sampling tests. The propellant shall be selected in accordance with 4.4.2.1 and subjected to the following tests as described under 4.5.

(a) Hydrogen peroxide assay	4.5.2
(b) Aluminum (Al)	4.5.4
(c) Chloride (Cl^-)	4.5.5
(d) Ammonium (NH_4^+)	4.5.6
(e) Nitrate (NO_3^-)	4.5.7
(f) Phosphate (PO_4^{3-})	4.5.8
(g) Sulfate (SO_4^{2-})	4.5.9
(h) Tin (Sn)	4.5.10
(i) Carbon (C)	4.5.11
(j) Evaporation residue	4.5.12
(k) Stability	4.5.13
(l) Particulate	4.5.14

4.4.2.1 Sampling plan.

4.4.2.1.1 Lot. A lot shall consist of the propellant produced by one manufacturer, with no change in process or material, in not more than 24 consecutive hours, provided the operation is continuous. In the event the process is a batch process, each batch shall constitute a lot.

4.4.2.1.2 Sample. Three one liter samples of propellant shall be selected as specified in 4.4.2.1.3, 4.4.2.1.4, and 4.4.2.1.5. Each sample taken shall be placed in a clean, clear, glass container and examined and tested in accordance with 4.5.1 and 4.5.2. The sample determined to have the lowest assay shall be subjected to additional tests in accordance with 4.5.3 through 4.5.13. Unless otherwise specified, quality conformance tests shall be made upon the samples of propellant taken directly from shipping containers at place of manufacture and time of shipment. When required, one liter samples shall be forwarded to a laboratory designated by the procuring activity for subjection to the quality conformance tests specified herein.

4.4.2.1.3 Drums. Unless otherwise specified, samples shall be drawn by means of an accepted sampling device. The number of drums selected from each lot shall be in accordance with MIL-STD-105, 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.

4.4.2.1.4 Drum filling. When specified by the contract or order (6.2), samples shall be taken during the process of filling drums from a storage tank. One sample shall be taken at the beginning, one when half the drums are filled and one while the last drum is being filled.

4.4.2.1.5 Tanks. Samples shall be taken by means of a sampling thief or other accepted device, from each cargo tank, tank car, or storage tank offered for delivery before drum filling. One sample shall be drawn within 1 foot of the surface, one near the mid-point, and one within 1 foot of the bottom.

4.5 Test methods.

4.5.1 Examination of product. Twenty-five milliliter (ml) specimens 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 distilled water to verify conformance with this specification as to clarity and color.

4.5.2 Hydrogen peroxide assay. The content of the sample shall be determined by the following method.

4.5.2.1 Procedure. Introduce about 10 drops of the propellant into a clean, tared weighing bottle, using a clean, dry, eye-dropper, then cover. Determine the sample weight accurately to the nearest 0.1 milligram. Carefully drop the weighing bottle (without cover) with its contents into a 500 ml Erlenmeyer flask containing 150 ml of five percent sulfuric acid solution. Add two drops of ferroin indicator and two drops of osmium tetroxide solution. Mix the contents and titrate with 0.5 N ceric sulfate until the salmon color of the indicator disappears. A reagent blank is titrated prior to the sample to the same colorless end point. A faint green or blue color will appear if overtitrated. Apply corrections to the burette reading for the blank. The titrant shall be at the same temperature during the assay and the standardization.

4.5.2.2 Calculation. The percent of H_2O_2 shall be calculated by the following formula:

$$H_2O_2, \text{ percent by weight} = \frac{\text{Corrected ml ceric sulfate} \times N \text{ ceric sulfate} \times 1.7}{\text{weight of sample in grams}}$$

4.5.2.3 Standardization of ceric sulfate solution. Weigh 0.3 to 0.5 g of arsenious oxide to the nearest 0.1 mg and transfer to a 500-ml Erlenmeyer flask. Add 20 to 30 ml of distilled water, 2 g of sodium hydroxide pellets, warm gently while stirring, until solution is complete; then add 100 ml of distilled water. Add 20 to 30 ml of 5 percent sulfuric acid. Add two drops of osmium tetroxide, two drops of ferroin indicator, and titrate with ceric sulfate solution until the salmon color disappears.

$$\text{Normality of ceric sulfate} = \frac{W \times a}{1.995 \times v}$$

Where: W = weight of arsenious oxide

a = assay of arsenious oxide (i.e. 99.5 wt%)

v = corrected volume of cer sulfate, ml

4.5.2.4 Reagents and equipment. The following reagents and equipment shall apply as test conditions 1.5.2:

(a) Reagents:

(1) Ceric sulfate solution 0.5 N. Add 294.2 g of ceric ammonium nitrate to 98 ml of concentrated sulfuric acid. Dilute, with stirring, to one liter, with distilled water.

(2) Osmium tetroxide solution 0.01 N. Add 0.25 g of osmium tetroxide to 100 ml of 0.1 N sulfuric acid.

(3) Ferroin indicator: 0.1 M (molar). Add 0.14 g of ferrous sulfate heptahydrate to 20 ml distilled water. Add 0.3 g of 0-phenanthroline monohydrate and dilute to 100 ml.

(4) Sulfuric acid solution 5 percent. Add 30 ml of concentrated sulfuric acid to 950 ml distilled water.

(5) Arsenious oxide: primary standard grade.

(6) Sodium hydroxide: pellets, ACS reagent grade.

(b) Equipment:

(1) Bottle: weighing

(2) Flask: Erlenmeyer, 500 ml capacity.

(3) Flask: Erlenmeyer, 100 ml capacity.

(4) Burette: 50-ml capacity

(5) Balance: analytical, 1 mg sensitivity.

4.5.3 Sample decomposition. Specified quantities of hydrogen peroxide will be decomposed and evaporated to dryness for each method of paragraphs 4.5.4 to 4.5.9 inclusive. The decompositions are performed by heating an acidic, alkaline, or neutral sample solution as follows.

(a) Acid decomposition: Place approximately 50 ml distilled deionized water in a large casserole and add 0.5 ml 1N sulfuric acid. Add a 5 ml to 10 ml increment of the specified peroxide volume. Place on a steam bath, cover with a ribbed watch glass, and allow to decompose. Control the reaction rate with additions of distilled water. Add 5 to 10 ml increments of peroxide until the specified volume has been decomposed.

(b) The decomposition of hydrogen peroxide with alkali is essentially the same as (a), except that 0.2 ml 1N sodium hydroxide is added to the casserole in place of the sulfuric acid. Proceed as outlined in (a).

(c) Decomposition without acid or alkali is accomplished by carefully evaporating a specified volume in the casserole, covered with a ribbed watch glass, and placed on the steam bath.

In (a), (b), or (c) an equal volume of distilled water is placed in a second evaporating dish, and treated in an identical manner. This is the reagent blank.

4.5.4 Aluminum. The sample shall be tested for aluminum (Al) content in accordance with the following method.

4.5.4.1 Procedure. Decompose a 25.0-ml sample as outlined in 4.5.3 (c). Rinse the watch glass with distilled water into the casserole and again evaporate to dryness. Add 2.0 ml of 2N hydrochloric acid and heat on the steam bath to dissolve the residue. Quantitatively transfer the contents of the casserole to a 50-ml volumetric flask. Add 1 ml of the 1 percent thioglycolic acid and 15.0 ml of aluminon-buffer, and dilute to the 50-ml mark with distilled water and mix. Place the flasks on a steam bath for 0.5 hour and allow to cool to room temperature for 0.5 hour in darkness. Obtain the absorbance of each sample in a 5-cm cell at 520 mμ after setting the blank to "0" absorbance.

4.5.4.2 Calculation. The aluminum content shall be calculated by locating the Al concentration of the sample on the calibration curve and using the following formula:

$$\text{Al, mg/l} = \frac{(\text{mg Al from calibration curve}) \times 1000}{\text{ml sample}}$$

4.5.4.3 Calibration curve. Transfer 1.0 ml, 2.0 ml, 4.0 ml, and 8.0 ml each of the dilute aluminum standard solution to four 50-ml volumetric flasks, using a microburette. A fifth 50-ml flask is to be used for a reagent blank. Add 2.0 ml of 2N hydrochloric acid and 10 ml of distilled water to each and mix the solution. Add 1 ml of the 1 percent thioglycolic acid and 15.0 ml of the aluminon-buffer, and dilute to the 50-ml mark with distilled water and mix. Place the flasks on a steam bath for 0.5 hour and allow to cool to room temperature for 0.5 hour in darkness. Obtain the absorbance of each standard in a 5-cm cell at 520 mμ after setting the blank at "0" absorbance. Plot the absorbance against mg Al, and draw the best fitting curve through the points. A new calibration curve is required for each fresh preparation of buffer solution. The absorbance of the sample is also obtained in a 5.0-cm cell and the Al content determined from the calibration curve.

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

(a) Reagents:

(1) Thioglycolic acid, 1 percent: Dilute 1 ml of high purity (95 percent or better) thioglycolic acid to 100 ml. This solution is unstable and should be made up fresh every few days.

(2) Hydrochloric acid solution, 2N: Dilute 43 ml of concentrated (36 percent) HCl to 250 ml with distilled water.

(3) Aluminon-buffer solution: Prepare by combining 335 ml of solution (a) with 335 ml of solution (b) and 335 ml of solution (c), each of which is prepared separately as follows:

Solution (a): Dissolve 4.0 g of gelatin in 50 ml of boiling distilled water, then dilute to 400 ml with distilled water.

Solution (b): Mix, with cooling, 190 ml ACS reagent grade ammonium hydroxide with 170 ml ACS reagent grade glacial acetic acid. Add more ammonium hydroxide or glacial acetic acid, as required, so that a 20 fold diluted test sampler has a pH of 5.25 to 5.35. Then add sufficient distilled water to the buffer to bring its final volume to 400 milliliters.

Solution (c): Dissolve 2.64 g of ACS reagent grade benzoic acid in 66 ml of ACS reagent grade methanol. Dissolve 0.35 g of aurin tricarboxylic acid in 190 ml of distilled water, to which are added the 66 ml of the benzoic acid-methanol solution. Mix and dilute to 335 ml with additional distilled water.

After solutions (a), (b), and (c) have been combined and mixed, allow 3 days aging prior to use. Store in a well stoppered brown bottle. Stability is about 6 months.

(4) Aluminum stock std. solution: Dissolve 3.534 g of aluminum potassium sulfate, $\text{Al K (SO}_4)_2 \cdot 12\text{H}_2\text{O}$, ACS reagent grade, in a 1 liter volumetric flask containing distilled water, and dilute to the mark (1 ml = 0.2 mg Al).

(5) Aluminum standard, dilute solution: Pipette exactly 5.0 ml of the stock standard and dilute to the mark in a 500 ml volumetric flask (1 ml = 0.002 mg Al). Make up fresh for each new calibration curve.

(6) Aluminum-free distilled water: This is distilled, deionized water, 100 ml of which has been evaporated, and when tested for aluminum, showed no increase over the reagent blank.

(b) Equipment:

- (1) Casserole: porcelain or teflon, 125-350 ml capacity.
- (2) Pipette: 10-ml capacity.
- (3) Watch glass: ribbed, 2 required.
- (4) Steam bath
- (5) Flask: volumetric, 50-ml capacity, 7 required.
- (6) Beaker: glass, 1-liter capacity, 2 required.
- (7) Beaker: glass, 500-ml capacity.
- (8) Burette: 0.01 ml subdivision, 25-ml capacity.
- (9) Bottle: glass, brown.
- (10) Spectrophotometer: capable of accommodating a 5-cm path cell.

4.5.5 Chloride. The sample shall be tested for chloride (Cl^-) content in accordance with the following method.

4.5.5.1 Procedure. This method is extremely sensitive and easily affected by dust, laboratory fumes, contaminated glassware, and impure reagents. Carefully evaporate and decompose a 50.0-ml sample on the steam bath as outlined in 4.3.3 (b). When evaporation and decomposition

are complete, wash the sides of the dish and the watch glass with chloride-free distilled water, and again evaporate to dryness. Dissolve the residue with 2.0 ml of ferric ammonium sulfate reagent and 2 ml of chloride-free distilled water. Quantitatively transfer the solution to a 25-ml volumetric flask. Rinse the dish at least twice and add the washings to the flask. Add 1.0 ml saturated mercuric thiocyanate reagent, mix by swirling, and dilute to the 25-ml mark with chloride-free distilled water. Mix again by inverting the flask several times and allow to stand in darkness for 15 to 30 minutes. Measure the absorbance of the reagent blank and sample solutions at 460 mμ in a 5.0-cm cell, after setting distilled water to "0" absorbance. Subtract the absorbance of the reagent blank from the sample absorbance. The chloride content is determined from the calibration curve constructed according to 4.5.5.3.

4.5.5.2 Calculation. The chloride content shall be calculated as follows:

$$\text{Cl}^-, \text{ mg/l} = \frac{(\text{mg Cl}^- \text{ from calib. curve}) \times 1000}{\text{ml sample}}$$

4.5.5.3 Calibration curve. A calibration curve shall be prepared as follows: Pipet 0, 1 ml, 2 ml, 4 ml, and 8 ml each of the dilute chloride standard solution (0.010 mg/ml) into five 25-ml volumetric flasks. Add 0.20 ml of 1N sodium hydroxide to each. Add 2.0 ml ferric ammonium sulfate and 5 ml chloride-free water and swirl. Add 1.0 ml saturated mercuric thiocyanate and mix. Add chloride-free distilled water to the 25-ml mark and mix by inverting several times. Place standards and blank in darkness for 15 to 30 minutes. Measure the absorbance of the blank and each of the four standards in a 5.00-cm cell at 460 mμ after setting distilled water to "0" absorbance. Subtract the absorbance of the blank from that of all the standards. Plot this difference against the corresponding mg of Cl^- .

4.5.5.4 Reagents and Equipment

(a) Reagents:

(1) Sodium hydroxide solution, 1N: Dissolve 40 g of ACS reagent grade sodium hydroxide pellets in sufficient chloride-free distilled water to make 1 liter of solution.

(2) Ferric ammonium sulfate: Mix 240 ml concentrated nitric acid and 160 ml of chloride-free distilled water. Dissolve 48.2 g of ferric ammonium sulfate, ACS grade, in the nitric acid solution and allow the precipitate to settle out. Decant the liquid for use in the analysis.

(3) Saturated mercuric thiocyanate: Saturate ACS reagent grade ethyl alcohol (95 percent) with ACS reagent grade mercuric thiocyanate. Allow the excess to settle out and decant the supernatant liquid for use in the analysis.

(4) Chloride stock solution: Dissolve exactly 165 mg (0.165g) of dried, primary standard grade, sodium chloride in chloride-free distilled water contained in a 100-ml volumetric flask and dilute to the mark (1.0 mg Cl/ml).

(5) Chloride standard solution: Pipet exactly 10.0 ml of the chloride stock solution into a 1000-ml volumetric flask, and dilute to the mark with chloride-free water (0.010 mg Cl/ml).

(6) Chloride-free water: This shall be double-distilled, or distilled deionized water, which tests negative (no cloudiness) with silver nitrate.

(b) Equipment:

(1) Evaporating dishes: Porcelain or teflon, 125 ml to 350 ml, as required.

(2) Watch glasses: ribbed.

(3) Pipettes: 1-, 2-, 4-, 8-, 10-, and 50-ml capacity.

(4) Burette: 25 ml, with 0.01 ml divisions.

(5) Volumetric flasks: 25-, 100-, and 1000-ml capacity.

(6) Reagent bottles as necessary.

(7) Steam bath.

(8) Analytical balance.

(9) Spectrophotometer or filter photometer: Single or double beam, capable of accommodating 1.0-cm to 5.0-cm cells.

4.5.6 Ammonium. The sample shall be tested for ammonium (NH_4^+) content in accordance with the following method.

4.5.6.1 Procedure. Decompose a 50.0-ml sample as outlined in 4.5.3 (a). Quantitatively transfer the contents of the sample and blank casseroles into separate 50-ml volumetric flasks, adding distilled water until the flasks contain approximately 40 ml of solution.

Add 0.5 ml 1N sodium hydroxide and 2.0 ml of Nessler's reagent. Dilute to the mark with distilled water and mix. Allow 30 to 60 min for color development. Measure the sample absorbance at 420 mμ in a 1.0 cm cell after setting the blank to "0" absorbance. The concentration, mg NH₄⁺, shall be obtained from the calibration curve, constructed according to 4.5.6.3.

4.5.6.2 Calculation. The ammonium content of the sample shall be calculated as follows:

$$\text{NH}_4^+, \text{ mg/l} = \frac{(\text{mg NH}_4^+ \text{ from calibration curve}) \times 1000}{\text{ml sample}}$$

4.5.6.3 Calibration curve. A calibration curve shall be prepared as follows: Transfer to 50-ml volumetric flasks exactly 1.0, 2.0, 4.0, 8.0 ml each of the dilute NH₄⁺ standard solution using a microburette. A reagent blank is prepared with 5 ml of distilled water in another 50-ml volumetric flask. Add 0.5 ml 1N sulfuric acid solution and 0.5 ml 1N sodium hydroxide solution. Add enough distilled water to bring the volume to approximately 40 ml. Add 2.0 ml Nessler's reagent, mix, dilute to the mark, mix again, and allow to stand 30 to 60 min. for color development. Measure the absorbance of the standards at 420 mμ in a 1.0-cm cell after setting the blank at "0" absorbance. Plot the absorbance vs. mg NH₄⁺ and draw a smooth curve through the origin. A 1 mμ to 2 mμ bandwidth at 420 mμ is necessary if conformity to Beer's law is desired.

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

(a) Reagents:

(1) Sulfuric acid, 1N: Dissolve 14.25 ml of concentrated (36 N) sulfuric acid in distilled water and dilute to 500 milliliters.

(2) Sodium hydroxide, 1N: Dissolve 20 g of ACS reagent grade sodium hydroxide in distilled water and dilute to 500 ml, boil for 5 minutes before use; store in a polyethylene bottle.

(3) Nessler's reagent: Dissolve 13 g of potassium iodide in 25 ml distilled water, using a 250-ml beaker. With constant stirring, add a cold saturated solution of mercuric chloride until the red precipitate formed no longer dissolves. Do not allow any mercuric chloride crystals to fall into the stirred solution. Carefully add a few crystals of potassium iodide to just dissolve the precipitate. Continue stirring and add 150 ml of a 14 percent potassium hydroxide

solution (25 g KOH in 150 ml). Allow to stand in a stoppered flask until clear. Decant the clear solution into a 250-ml volumetric flask and dilute to the mark with distilled water. Transfer to a brown reagent bottle, add by drops a saturated solution of mercuric chloride until a slight yellow permanent precipitate is formed. Mix, and keep well stoppered in darkness.

(4) Ammonium stock standard solution: Dissolve 1.486 g of dried ammonium chloride, ACS reagent grade, in distilled water and dilute to the mark in a 500-ml volumetric flask, (1 ml = 1.0 mg NH_4^+).

(5) Dilute ammonium standard solution: Pipette exactly 5.0 ml into a 250 ml volumetric flask and dilute to the mark with distilled water (1 ml = 0.020 mg NH_4^+).

(6) Distilled water: For the determination of NH_4^+ the water is to be distilled/deionized ammonia-free water.

(b) Equipment:

- (1) Casserole: porcelain or teflon, 125-ml to 350-ml capacity.
- (2) Pipette: 10- and 100-ml capacity.
- (3) Watch glass: ribbed, 2 required.
- (4) Steam bath.
- (5) Flask: volumetric, 50-ml capacity, 8 required.
- (6) Burette: 0.01 ml subdivision, 5-ml capacity.
- (7) Beaker: glass, 100- and 400-ml capacity.
- (8) Flask: volumetric, 1-liter capacity, 2 required.
- (9) Beaker: glass, 1-liter capacity.
- (10) Bottle: glass, brown.
- (11) Bottle: polyethylene, 200- and 500-ml capacity.
- (12) Stirrer: glass rod.
- (13) Spectrophotometer: capable of accommodating a 1-cm path cell.

4.5.7 Nitrate. The sample shall be tested for nitrate (NO_3^-) content in accordance with the following method.

4.5.7.1 Procedure. Decompose a 10-ml sample as outlined in 4.5.3 (b). Add 5 ml of distilled water and re-evaporate to dryness. Add 2.0 ml of phenoldisulfonic acid solution and heat on the steam bath for 15 minutes. Cool to ambient, add 10 ml of distilled water. Slowly add 10 ml of concentrated ammonium hydroxide, and mix. Quantitatively transfer to 50 ml volumetric flasks and dilute to the mark with distilled water and mix. Measure the absorbance of the sample at 410 m μ in a 5.0-cm cell after setting the blank at "0" absorbance. The NO_3^- content of the sample is obtained from the calibration curve constructed according to paragraph 4.5.7.3.

4.5.7.2 Calculation. The nitrate content of the sample shall be calculated as follows:

$$\text{NO}_3^-, \text{ mg/l} = \frac{(\text{mg NO}_3^- \text{ from calibration curve}) \times 1000}{\text{ml sample}}$$

4.5.7.3 Calibration curve. A calibration curve shall be prepared as follows: Transfer to 5 evaporating dishes exactly 0.5 ml, 1.0 ml, 2.0 ml, 4.0 ml, and 8.0 ml each of the dilute nitrate standards solution, using a microburette. A reagent blank is prepared with 5.0 ml of distilled water in another evaporating dish. Add 0.2 ml of 1N sodium hydroxide to all, and evaporate to dryness on the steam bath. Add 5 ml distilled water and again heat on the steam bath for 15 minutes. Cool, add 10 ml distilled water and 10 ml of ammonium hydroxide and mix. Quantitatively transfer the solutions to 50-ml volumetric flasks. Dilute to the mark with distilled water and mix. Measure the absorbance of the yellow solutions at 410 m μ in a 5.0-cm cell after setting the reagent blank at "0" absorbance. Plot the mg NO_3^- versus absorbance and draw a smooth curve (line) through the origin.

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

(a) Reagents:

(1) Sodium hydroxide solution: 1N. Dissolve 40.0 g of sodium hydroxide ACS reagent grade in 1 liter of distilled water and store in a polyethylene bottle.

(2) Phenoldisulfonic acid solution: Dissolve 50.0 g of phenol ACS reagent grade in 300 ml of concentrated sulfuric acid. Cautiously, add 75 ml of fuming sulfuric acid (30 wt. percent SO_3) and heat at 212°F (100°C) for 2 hours. Cool to ambient temperature. Transfer the solution into a brown glass bottle and store in a cool area. Before use, age for 24 hours.

(3) Ammonium hydroxide: concentrated (28 wt percent).

(4) Nitrate standard stock solution: Dissolve exactly 1.631 g ACS reagent grade potassium nitrate in distilled water contained in a 1 liter volumetric flask and dilute to the mark (1 ml = 1.0 mg NO_3^-).

(5) Nitrate dilute standard solution: Dilute 5.0 ml of the stock standard solution to 500 ml with distilled water (1 ml = 0.01 mg NO_3^-).

(b) Equipment:

(1) Casserole: porcelain or teflon, 125 ml to 350 ml capacity.

(2) Pipette: 10-, 15-, and 100-ml capacity.

(3) Watch glass: ribbed, 2 required.

(4) Steam bath.

(5) Flask: volumetric 50-ml capacity, 8 required.

(6) Burette: 0.01 ml subdivision, 25-ml capacity.

(7) Beaker: glass, 1-liter capacity, 3 required.

(8) Bottle: polyethylene, 1-liter capacity.

(9) Beaker: glass, 500-ml capacity.

4.5.8 Phosphate. The sample shall be tested for phosphate (PO_4^{3-}) content in accordance with ASTM Method F61-65T.

4.5.9 Sulfate. The sample shall be tested for sulfate (SO_4^{2-}) content in accordance with the following method.

4.5.9.1 Procedure. Decompose a 25-ml sample as outlined in 4.5.3 (b). Transfer the residue quantitatively to a 50-ml volumetric flask, using several 1 to 2 ml rinsings. Add 10.0 ml of 0.5 N hydrochloric acid and mix. Add 10.0 ml of the glycerin-alcohol solution,

dilute to the mark with distilled water and mix. Pour the solution into a clean, dry 150-ml beaker and add 0.3 g of barium chloride crystals. Immediately stir the solution for 1 minute at a constant rate with a glass rod or a magnetic stirrer. A reagent blank is prepared concurrently with the sample, except that the barium chloride crystal addition is omitted. Measure the absorbance at 420 mμ in a 5.0-cm cell with the blank set at "0" absorbance. Measure the absorbance after 20 minutes following the 1 minute stirring step.

4.5.9.2 Calculation. The sulfate content of the sample shall be calculated as follows:

$$\text{SO}_4^{-2} = \frac{(\text{mg SO}_4^{-2} \text{ from calibration curve}) \times 1000}{\text{ml Sample}}$$

4.5.9.3 Calibration curve. Transfer 2.0 ml, 4.0 ml, 6.0 ml, and 8.0 ml of the dilute standard sulfate solution to 50 ml volumetric flasks using a microburette. A reagent blank is prepared with 10.0 ml of distilled water in another 50-ml volumetric flask. Add 0.2 ml of 1N sodium hydroxide and 10.0 ml of 0.5 N hydrochloric acid and mix. Add 10.0 ml of the glycerin-alcohol solution and dilute to the mark with distilled water and mix. Pour the contents of each 50 ml flask containing a standard into clean, dry, 150-ml beakers, and add 0.3 g of barium chloride crystals. Immediately stir the solutions for 1 minute at a constant speed with a glass rod or magnetic stirrer. Measure the absorbance of each standard at 420 mμ in a 5.0-cm cell after setting the blank at "0" absorbance. The turbidity measurements are made after 20 minutes following the 1 minute stirring step. Plot the absorbance vs mg of sulfate and draw a smooth curve through the origin.

4.5.9.4 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.9:

(a) Reagents:

(1) Sodium hydroxide solution: 1 N: Dissolve 40.0 g of sodium hydroxide ACS reagent grade in 1 liter of distilled water and store in a polyethylene bottle.

(2) Hydrochloric acid solution, 0.5N: Dilute 42.8 ml of concentrated HCl to 1 liter with distilled water.

(3) Glycerin-alcohol-salt solution: Mix 50 ml ACS reagent grade glycerin with 300 ml distilled water. Add 75 g ACS reagent grade sodium chloride and 100 ml 95 percent ethyl alcohol or isopropyl alcohol, and mix.

(4) Barium chloride dihydrate ACS reagent grade: Assemble the sieves conforming to RR-S-366 with a 20-mesh size on top and a 30-mesh size on the bottom. Screen barium chloride through the sieves and collect the crystals retained on the 30-mesh for use.

(5) Sulfate standard solution: Dissolve exactly 0.740 grams of anhydrous sodium sulfate, ACS reagent grade in distilled water, and dilute to the mark in a 500-ml volumetric flask (1 ml = 1.0 mg SO_4^{2-}).

(6) Sulfate dilute standard solution: Pipet exactly 5.00 ml of the stock standard solution into a 500-ml volumetric flask and dilute to the mark with distilled water (1 ml = 0.010 mg SO_4^{2-}).

(b) Equipment:

(1) Casserole: porcelain or teflon, 125-ml to 350-ml capacity.

(2) Pipette: 25- and 50-ml capacity.

(3) Steam bath.

(4) Flask: volumetric, 50-ml capacity, 8 required.

(5) Beaker: glass, 150-ml capacity, 9 required.

(6) Stirrer: glass rod.

(7) Burette: 0.01 subdivision, 25-ml capacity.

(8) Beaker: glass, 1-liter capacity, 2 required.

(9) Bottle: polyethylene, 1-liter capacity.

(10) Sieve: 20- and 30-mesh size.

(11) Flask: volumetric, 500-ml and 1-liter capacity.

(12) Spectrophotometer: capable of accommodating a 5-cm path cell.

4.5.10 Tin. The sample shall be tested for Tin (Sn) content in accordance with ASTM Method F62-65T.

4.5.11 Carbon. The sample shall be tested for carbon (C) content in accordance with the following procedure.

4.5.11.1 Procedure. The test for carbon content in the sample shall be conducted using the apparatus prepared and assembled in accordance with figures 1 and 2. All operations for this procedure should be conducted in an atmosphere free of organic vapors, air, CO_2 , and any organic material. The reaction flask must be cleaned thoroughly after each determination. Two washes with nitric acid or heating with chromic acid are necessary. A standard sample shall be run to determine the efficiency of the collection flask.

4.5.11.1.1 Decomposition of the sample. Set up the decomposition assembly as shown in figure 1. Add 50 ml distilled water. Transfer by pipette to the clean reaction flask, a 50-ml aliquot of the hydrogen peroxide sample. Always use a suction bulb on the pipette. With the West type condenser in place, add 5 ml of 0.08N sodium hydroxide solution. Circulate cold water through the condenser. Close the stopcock of the addition funnel and close the third neck of the flask with a ground glass stopper. Lubricate joints with distilled water. Place in ice bath and then add one drop of the dilute silver nitrate solution to the sample through the condenser tube. When the reaction has ceased, add a second drop. Continue in this manner until the decomposition is complete. Small portions (1-2 ml) of distilled water may be used occasionally to wash silver nitrate droplets, adhering to the walls of the condenser, into the flask. The silver nitrate additions must be so slow that no water vapors escape from the top of the condenser. When the decomposition is complete, add 10 ml of the 10 wt percent silver nitrate solution through the addition funnel. Follow this by two additions of 5 ml each of the 9N sulfuric acid. Wash down the inside of the condenser with 40 ml of this sulfuric acid solution, followed by a small volume of distilled water. This operation may require 2 1/2 hours depending on the concentration of hydrogen peroxide.

4.5.11.1.2 Oxidation and determination. Remove the condenser and stopper from the reaction flask containing the decomposed sample, cool the solution and the flask and then connect the flask to the oxidation and absorption apparatus as shown in figure 2. Circulate cold water through the Friederichs condenser. Heat the furnace containing the cupric oxide tube to 1382°F (750°C) and maintain this temperature with a variable transformer. Draw CO - and CO_2 - free oxygen (MIL-P-25508D, Type I) through the apparatus for 5 minutes. Add 50 ml of $\text{Ba}(\text{OH})_2$ solution to the collection flask. Draw air through the train at a rate of 60-90 bubbles per minute and turn on full heat under the reaction flask. When the contents of the flask reach about 158 - 176°F (70 - 80°C), add a 100-ml portion of persulfate solution through the addition funnel. Maintain the bubbling rate through the collection flask by adjusting the screw clamp on the tube to the aspirator. Add two more 100-ml portions

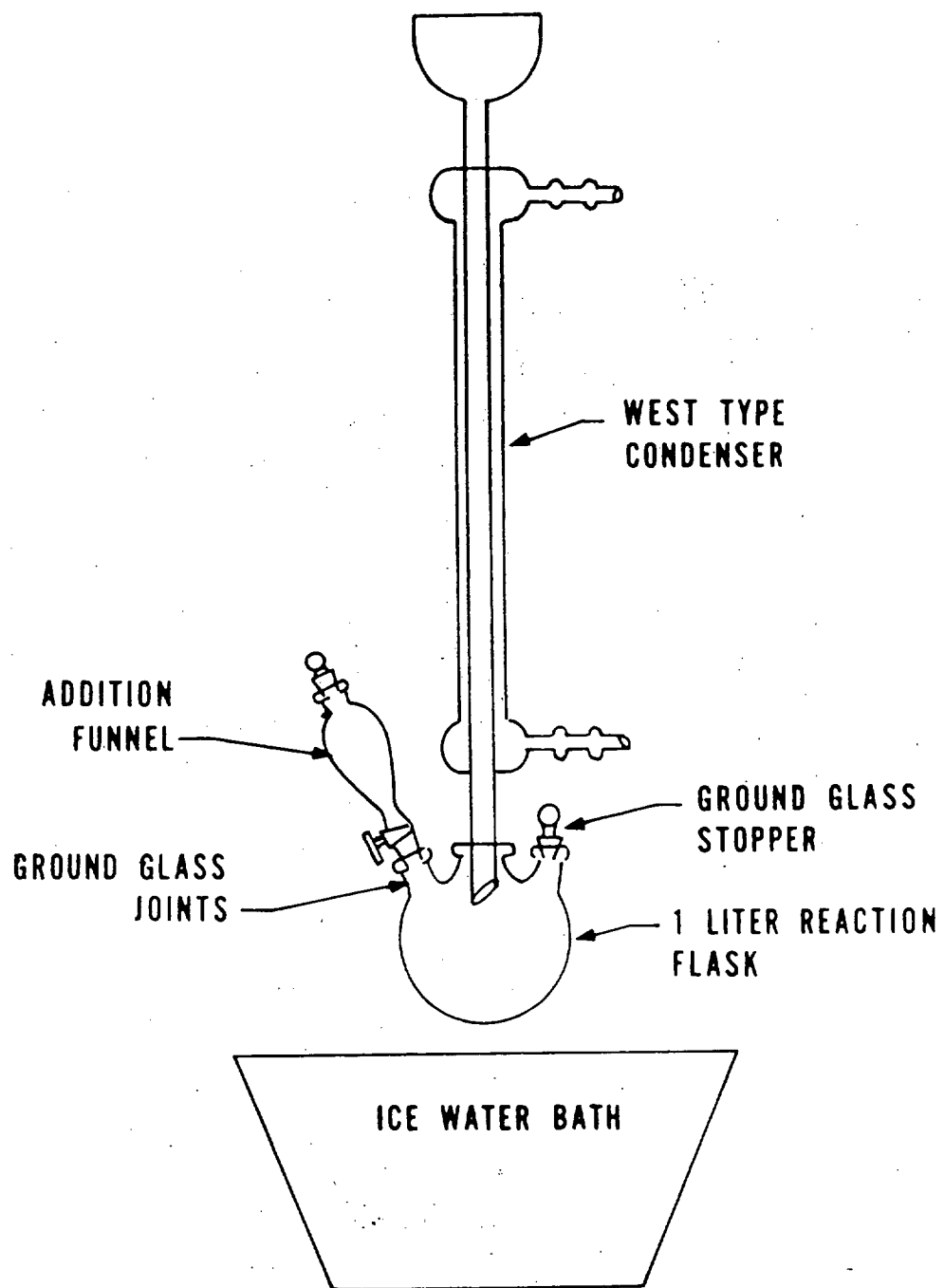


Figure 1. Decomposition Assembly

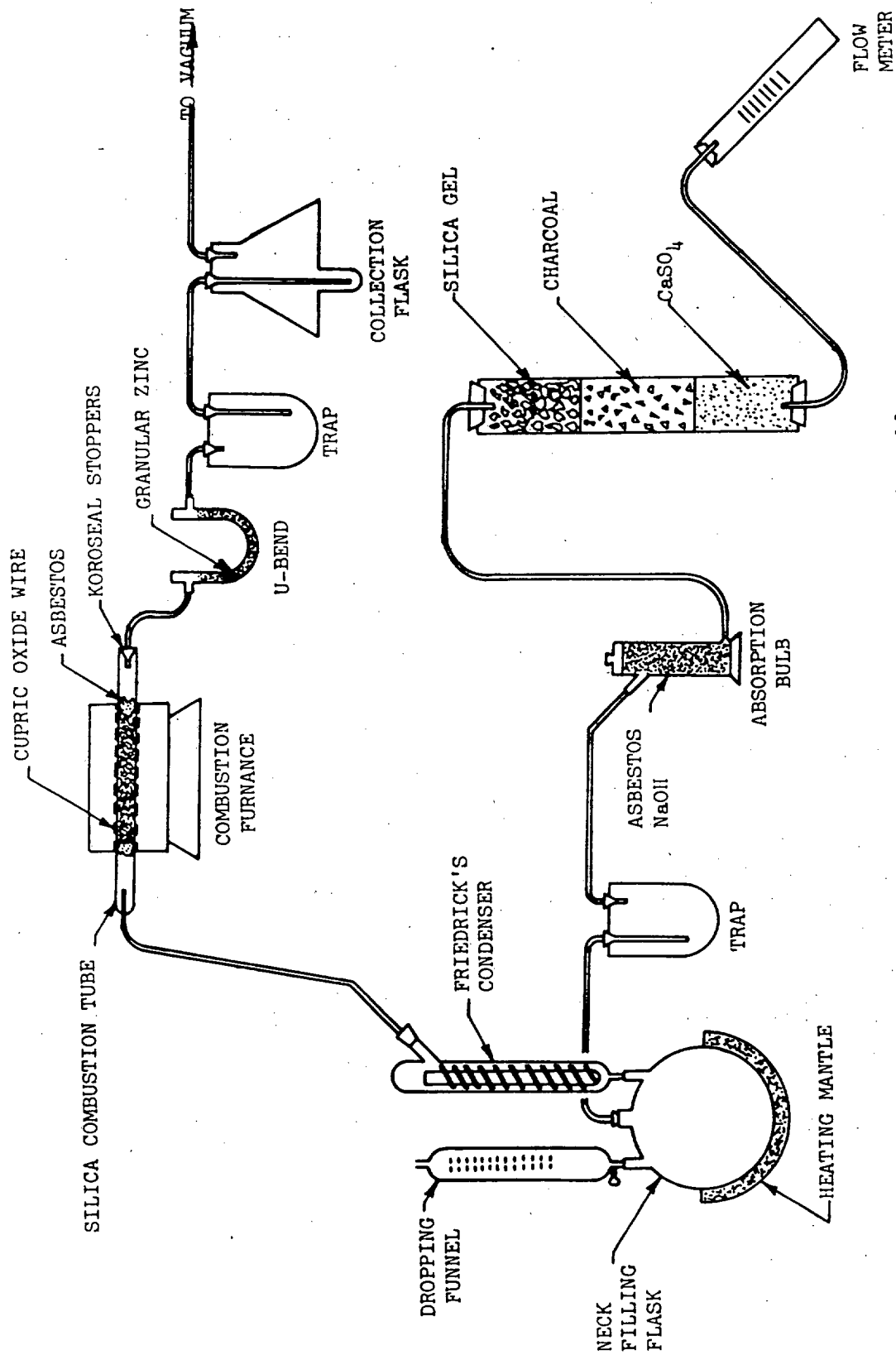


Figure 2. Oxidation and Absorption Assembly

of persulfate solution, each after the brown color due to the previous additions has faded. When the final brown color has faded, remove the solution, heating mantle, and open the stopcock on the addition funnel. Remove the collection flask and wash the bubbler tube, inside and out, into the flask with CO_2 -free distilled water. Add 3 drops of phenolphthalein indicator to the solution in the collection flask and mix thoroughly. Titrate immediately with standard hydrochloric acid to the disappearance of the pink color.

4.5.11.1.3 Blank. Run in the same manner as the H_2O_2 except substitute 50 ml of CO_2 -free distilled water for the H_2O_2 sample.

4.5.11.2 Calculation. The carbon content shall be calculated using the following formula:

$$\text{mg C/l} = \frac{(B-A) \times 6.0 \times (\text{N of HCl}) \times 1000}{\text{ml of sample}}$$

Where: A = ml HCl of blank

B = ml HCl for H_2O_2

4.5.11.3 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.11.

(a) Reagents:

(1) Barium hydroxide ($\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$), 100 grams per liter: Dilute 30 ml to 130 ml with CO_2 -free distilled water.

(2) Hydrochloric acid, 0.15 N to 0.20 N standardized against primary standard sodium carbonate.

(3) Potassium persulfate, saturated solution: Prepare a saturated solution of potassium persulfate, $\text{K}_2\text{S}_2\text{O}_8$, as needed.

(4) Silver nitrate, 10 wt percent: Dissolve 100 grams of silver nitrate in distilled water and dilute to 1 liter.

(5) Silver nitrate, dilute solution. Add 1 drop of 100 g/l silver nitrate solution to 10 ml of distilled water (CO_2 -free).

(6) Sodium hydroxide, 0.08 N: 8 g ACS reagent grade in 250 ml distilled water.

(7) Sulfuric acid solution, 9 N. Carefully add 250 ml of concentrated sulfuric acid (36N) to approximately 750 ml CO_2 -free distilled water with cooling. Dilute to 1 liter when cool.

(8) Phenolphthalein indicator. Dissolve 0.05 g phenolphthalein in 95 percent alcohol and dilute to 100 ml with 95 percent alcohol.

(b) Equipment:

- (1) Decomposition assembly (figure 1).
- (2) Oxidation and absorption assembly (figure 2).

4.5.12 Evaporation residue. The sample shall be tested for evaporation residue content in accordance with the following method.

4.5.12.1 Procedure. Add several pieces of clean platinum, having a surface area equivalent to the inside of two 100 ml casseroles, into a clean 2-liter pyrex beaker containing 500 ml of distilled water. Cover with a clean ribbed watch glass and place 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 with a clean ribbed watch glass. Add a small piece of clean platinum to the solution to prevent concentration of undecomposed sample. Boil the solution until condensed to a 25- to 50-ml volume. Discontinue boiling and transfer the remaining solution to a 100-ml platinum evaporating dish which has attained constant weight by heating. Allow the solution to completely evaporate. While decomposing and condensing the sample, add 500 ml of distilled water to a clean 1-liter pyrex boiling flask and cover with clean ribbed watch glass. Place on the 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 which has attained constant weight by heating. Place the dishes in a vacuum oven at 221° to 230°F (105° to 110°C) for a minimum of 1 hour. Remove the dishes and allow to cool in a desiccator. Place on a balance and record the weight to the nearest 0.1 milligram.

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

$$\text{Evaporation residue, mg/l} = \frac{\text{mg (corrected)}}{0.3}$$

4.5.12.3 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.12.

(a) Reagents:

Water: double distilled or deionized.

(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.

4.5.13 Stability. The sample shall be tested for stability in accordance with the following method.

4.5.13.1 Procedure. Transfer 50 ml of the sample into each of three 50-ml pyrex volumetric flasks which have been specifically prepared in accordance with 4.5.13.3. Cover the tops of the flasks immediately with a 5 to 10-ml pyrex beaker across the mouth of the flask 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 entering above the steam seal 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 plus or minus 0.01 gram.

4.5.13.2 Calculation. The percent loss of active oxygen shall be calculated by averaging the results of the individual flasks using the following formula:

$$\text{Percent active O}_2 \text{ loss} = \frac{W_1 - W_2}{CW_1 \times 0.470} \times 100$$

Where: W_1 = initial net weight.

W_2 = final net weight.

C = weight fraction sample (concentration/100).

4.5.13.3 Test flask preparation. Prepare the test flask by drying, and with aluminum cover, place on a balance. 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 sample. Flasks which gave inconsistent results shall be rejected. Flasks which are not rejected shall be used only to contain sample.

4.5.13.4 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.13.

(a) Reagents:

Nitric acid: concentrated, ACS reagent grade.

(b) Equipment:

(1) Flask: volumetric, pyrex, 50-ml capacity, 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.

4.5.14 Particulate. The sample shall be tested for contamination in accordance with ASTM D-2276-65T, Method A, with the exceptions noted below.

4.5.14.1 Exceptions. The following shall modify the above cited Method A.

(a) Disc, filter: solvent resistant teflon[®] polytetrafluoroethylene, plain white, 10 ± 3 pore size, 47 mm diameter.

(b) Volume of hydrogen peroxide to be filtered: 500 ml, collected in a clean, dry, glass bottle.

(c) Omit all organic solvents (alcohol, petroleum ether).

(d) Oven temperature for drying the filter disc must be 158°F (70°C).

4.6 Preparation for delivery inspection. The preservation, packaging, packing, and marking for shipment and storage of the propellant shall be inspected to determine compliance with the requirements of section 5 of this specification.

4.7 Rejection. When any sample of the propellant fails to conform to the requirements specified herein, the entire lot represented shall be rejected.

5. PREPARATION FOR DELIVERY

5.1 Packaging. The propellant shall be shipped in the unit quantities and packaged in drums, cargo tanks, or tank cars as specified by the procuring activity (6.2). All containers shall comply with the requirements of the Department of Transportation (DoT) Regulations 49 CFR 171-190. The contractor shall assure that all gaskets are serviceable and furnish new gaskets when necessary. The contractor shall perform inspection and cleaning to assure that all containers are free from contamination, and are suitable for shipment and storage.

5.1.1 Drums. Drums shall conform to DoT Specification 42D, aluminum of not over 55 gallon capacity with a vented closure in the top head. The closure shall be wire sealed to prevent removal in transit. Side closures shall not be permitted.

5.1.2 Cargo tanks. Cargo tanks shall conform to DoT Specifications MC 310-H₂O₂ or MC 312-H₂O₂ of types 1060, 1260, 5254 or 5652 aluminum alloy with approved venting and pressure relief devices approved by the Bureau of Explosives.

5.1.3 Tank cars. Tank cars shall conform to DoT Specification 103A-AL-W-H₂O₂ of types 1060, 1260, 5254, or 5652 aluminum alloy with venting arrangement approved by the Bureau of Explosives.

5.2 Labeling and marking. Each container and shipment shall be labeled and placarded in accordance with DoT Regulations 49 CFR 171-190. Other markings shall be in accordance with MIL-STD-129. In addition, drums shall be marked in accordance with MIL-STD-172.

5.2.1 Identification tag. An identification tag impervious to climatic conditions shall be wired to the closure or discharge of each container and shall contain the following information.

Propellant _____ (name) _____

Specification _____ MIL-P-16005 _____

Type _____

FSN No. _____ 9135 _____

Quantity _____

Cont. or Order No. _____

Name of Manufacturer _____

Name of Contractor _____ (if different from mfg.)

Date of Manufacture _____

6. NOTES

6.1 Intended use. The propellant covered by this specification is intended for use as an oxidizer or mono-propellant in rocket engines.

6.2 Ordering data. Procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Type of propellant (Type I or Type II).
- (c) Method of shipment, type and capacity of containers.
- (d) Quantity by weight in pounds (avoirdupois).
- (e) Drum filling special provisions (4.4.2.1.4).

(f) That two copies of the test report, signed by the Contractor's representative, listing values obtained on all tests (quantitative values where method provides) shall accompany each shipment delivered to the consignee. In addition, one copy should also be furnished the AFRPL (RPPS), Edwards, California 93523.

6.3 Definition.

6.3.1 Particulate. The undissolved solids retained on a 10-micron filter membrane.

6.4 Control filter tare weight. An increase in tare weight of the control filter is an indication of improper water rinsing.

Custodians:

Army - MI
Navy - WP
Air Force - 12

Preparing activity:

Air Force - 12
Project No. 9135-0039

Reviewers:

Air Force - 19, 68

Other interests:

NASA

Reviewer/user information is current as of the date of this document. For further coordination of changes to this document, draft circulation should be based on the information in the current DoD Index of Specifications and Standards.

SPECIFICATION ANALYSIS SHEET		Form Approved Budget Bureau No. 119-R004
INSTRUCTIONS		
<p>This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity (as indicated on reverse hereof).</p>		
SPECIFICATION		
MIL-P-16005E Propellant, Hydrogen Peroxide		
ORGANIZATION (of submitter)		CITY AND STATE
CONTRACT NO.	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT \$
MATERIAL PROCURED UNDER A		
<input type="checkbox"/> DIRECT GOVERNMENT CONTRACT <input type="checkbox"/> SUBCONTRACT		
1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?		
A. GIVE PARAGRAPH NUMBER AND WORDING.		
B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES.		
2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID		
3. IS THE SPECIFICATION RESTRICTIVE?		
<input type="checkbox"/> YES <input type="checkbox"/> NO IF "YES", IN WHAT WAY?		
4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)		
SUBMITTED BY (Printed or typed name and activity)		DATE