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MILITARY SPECIFICATION

HYDROGEN PEROXIDE, E-STABILIZED, 70% AND 90% (FOR TORPEDO USE)

This specification has been approved by the Bureau of Naval Weapons, Department of the Navy.

1. SCOPE

1.1 This specification covers aqueous solutions of hydrogen peroxide in which additives have been dissolved to impart a low decomposition rate, to repress the effects of accidental catalytic contamination and to prevent corrosion of aluminum containers. Such hydrogen peroxide solutions are designated as "E" stabilized.

1.2 Classification - E-stabilized hydrogen peroxide shall be of the following grades as specified (see 6.2)

Grade 70E- 70% hydrogen peroxide by weight
Grade 90E- 90% hydrogen peroxide by weight

2. APPLICABLE DOCUMENTS

2.1 The following specification and standards, of the issue in effect on date of invitation for bids, form a part of this specification:

SPECIFICATION

Military

MIL-A-799	- Aluminum, High-Purity, Wrought
MIL-STD-105	- Sampling Procedures and Table for Inspection by Attributes
MIL-STD-129	- Marking for Shipment and Storage

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

2.2 Other Publications - The following publication forms a part of this specification. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

NAVY DEPARTMENT, BUREAU OF NAVAL WEAPONS

NAVORD Report 543 - Navol Loading Depot
NAVORD Report 5216 - Introduction to Hydrogen Peroxide

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

INTERSTATE COMMERCE COMMISSION

Regulations for Transportation of Explosives and other Dangerous Articles, etc.

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington 25, D. C.)

3. REQUIREMENTS

3.1 Material - The E-stabilized hydrogen peroxide shall be clear, colorless solution of hydrogen peroxide and additives in water.

3.2 Concentration - The concentration (70% or 90%) shall be specified by the bureau or agency concerned, and shall be designated as the percent hydrogen peroxide by weight contained in the solution. Unless otherwise specified, this concentration shall not vary by more than plus 1.0 or minus 0.5 percent.

3.3 Additives - E-stabilized hydrogen peroxide solutions shall contain the following constituents:

Tin (Sn)	32 $\pm$ 4 milligrams per liter
Phosphate (PO_4^{---})	29 $\pm$ 4 milligrams per liter
Nitrate (NO_3^-)	110 $\pm$ 20 milligrams per liter

respectively introduced in 70% or 90% hydrogen peroxide by use of the following additives:

Sodium stannate ($Na_2SnO_3.3H_2O$)

Sodium phosphate, Dibasic, Dodecahydrate ($Na_2HPO_4.12H_2O$)

Sodium nitrate ($NaNO_3$) and Nitric acid (HNO_3) for pH adjustment

3.4 Objectionable Impurties - One liter of E-Stabilized hydrogen peroxide solution shall contain no more than

1 milligram of Chloride (Cl^-) and 10 milligrams of Sulfate (SO_4^{--}) when tested in accordance with 4.5.6 and 4.5.7 respectively.

3.5 Evaporation Residue - The evaporation residue, when tested as specified in 4.5.8 shall not exceed 325 milligrams per liter.

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3.6 Stability - E-stabilized hydrogen peroxide solutions shall not lose more than 2.0 percent (maximum) of its original active oxygen when tested according to 4.5.9.

3.7 Acidity, pH - Unless otherwise requested by the contractor and agreed to by the purchasing activity, the apparent pH of E-stabilized hydrogen peroxide solutions, determined as specified in 4.5.10 shall be:

1.4 $\pm$ 0.2 for Grade 70 E

0.2 $\pm$ 0.2 for Grade 90 E

If necessary, pH adjustment is made by the use of either pure nitric acid, or pure sodium hydroxide solutions in pure water.

3.8 Surface Tension - Unless otherwise requested by the contractor and agreed to by the purchasing activity, the surface tension of E-stabilized hydrogen peroxide solutions when determined by the method of 4.5.11 shall be no less than the following values:

	Minimum Surface Tension (dynes/centimeter)
Grade 70 E	72 at 20°C
Grade 90 E	74 at 20°C

4. QUALITY ASSURANCE PROVISIONS

4.1 The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. 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.

4.2 Sampling[1]

4.2.1 Lot - For purposes of sampling, a lot shall consist of all material of the same type offered for delivery at the same time in drums filled from one storage tank at one time.

4.2.1.1 When offered in tank car or storage tank - For purposes of sampling, a lot shall consist of all material of the same type, stored in one storage tank, and offered for delivery at the same time.

[1]All glassware and equipment for purposes of sampling shall be properly cleaned and passivated prior to use.

ative measurements made before and 4.3.6.6 Reference measurements. When requirements are based on compar

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4.2.1.2 Time of Sampling - Sampling shall be accomplished not earlier than three weeks prior to shipping date.

4.2.2 Sampling for Inspection of filled Containers - A random sample of filled containers shall be selected from each lot by the inspector in accordance with Standard MIL-STD-105 at Inspection Level I and acceptable quality level (AQL) equal 2.5 percent defective to verify compliance with this specification regarding fill, closure, marking, and other requirements not involving tests.

4.2.3 Sampling for Lot Acceptance Tests

4.2.3.1 Sampling from tank cars or from storage tank (before drums are filled). - From each tank car or storage tank of hydrogen peroxide offered for delivery three 1-qt samples shall be taken by means of a sampling thief or other suitable device, for testing. One sample shall be drawn approximately one foot from the bottom of the tank and one from approximately one foot below the surface of the liquid. The third shall be taken midway between the top and the bottom. Each sample shall be placed in a clean, clear glass container and inspected as specified in 4.5.1 and 4.5.2. The one of the three specimens which shows the lowest assay shall be subjected to the tests specified in 4.5.3 through 4.5.11.

4.2.3.2 Sampling during the filling of drums - When the inspector is present while the drums are being filled from a storage tank, three one-quart samples shall be taken during the filling process. One sample shall be taken at the beginning, one when the tank is half empty and one while the last drum is being filled. Each sample shall be placed in a clean, clear, glass container and inspected as specified in 4.5.1 and 4.5.2. The one of the three specimens which shows the lowest assay shall be subjected to the tests specified in 4.5.3 through 4.5.11.

4.2.3.3 Sampling from drums - From each sampling lot of hydrogen peroxide offered for inspection in drums, a number of drums shall be selected at random in accordance with Table I. From each sample drum, a one-liter specimen shall be drawn by means of an accepted sampling device. The specimen shall be taken approximately one inch below the surface of the liquid. Each specimen shall be placed in a separate clean, dry, glass container, and labeled for identification of the container and the lot represented. Each of the samples shall be tested in accordance with 4.5.1 and 4.5.2 and the sample showing the lowest assay shall be subjected to the tests specified in 4.5.3 through 4.5.11.

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Table I. Sampling procedure (drums)

Number of drums in the lot	Number of drums in sample	Acceptance number (defectives)	Rejection number (defectives)
15 and under	2	0	1
16 to 40	3	0	1
41 to 65	5	0	1
66 to 110	7	0	1
111 and over	10	0	1

4.3 Inspection

4.3.1 Inspection of filled containers - Each sample filled container selected in accordance with 4.2.2 shall be examined by the inspector for defects of the container and the closure for evidence of leakage, and for unsatisfactory markings; each sample filled container 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 inspector has evidence that these may not be correct. The inspector shall place his hand on each of the sample drums to detect any unusual temperature; and his ear to detect any sound of activity within the drum; these are signs of decomposition of the hydrogen peroxide, and any drum showing either is defective. Any container in the sample having one or more defects, or under required fill, shall be rejected and if the number of defective containers in any sample exceeds the acceptance number for the appropriate sampling plan of Standard MIL-STD-105, the lot represented by the sample shall be rejected.

4.4 Lot Acceptance Tests - Samples selected in accordance with 4.2.3 shall be subjected to the tests specified in 4.5. Lots shall be accepted or rejected on the basis of the laboratory test results of these samples.

4.4.1 Time of Testing - Testing shall be performed not later than four weeks after the sampling date.

4.4.2 Action in case of failure - If any one of the samples tested is found to be not in conformance with this specification, the lot which it represents shall be rejected.

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4.4.3 Reports on Inspection and Tests - Two copies of each lot acceptance inspection and test report shall be forwarded to the procuring activity and the Bureau of Ordnance by the inspector. The inspection and test report shall certify the results of the required determination, the dates of sampling, test and shipment, and shall include a brief description of the test methods employed, in case any deviation from these specified under 4.5 have been adopted.

4.5 Test Procedures[1]

4.5.1 Visual Inspection - Twenty-five milliliter (ml) specimens shall be taken from each of the samples and shall be placed in separate 8 inch by 1 inch 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 by the inspector to verify conformance with this specification as to clarity and color.

4.5.2 Concentration

4.5.2.1 Reagent - standard $KMnO_4$ solution. - Make up an approximately 0.5N solution of $KMnO_4$ and age in the dark for at least one week. Filter the solution through pure asbestos (free from organic matter), or through a medium-porosity sintered glass crucible before standardizing against Bureau of Standards sodium oxalate. Weigh an approximately 1.5 gram sample of sodium oxalate which has been dried for 1 hour at 105° to 110° C. Dissolve the oxalate in 250 ml of 1:3 sulfuric acid, previously boiled for 10 to 15 minutes and then cooled. Add from the calibrated burette 90 to 95 percent of the necessary $KMnO_4$, (25° C.), and let stand until the pink color disappears. Heat to 55° to 60°C and complete the titration by adding the permanganate dropwise, allowing each drop to become decolorized before the next is added. The end point shall be taken at the point where the pink color persists for 30 seconds. Determine the reagent blank by titrating the same volume of boiled and cooled sulfuric acid with the permanganate. Subtract the volume required for the blank from the original titration.

Calculation:

$$\text{Normality } KMnO_4 = \frac{\text{Weight sodium oxalate} \times \text{purity}}{0.0670 \times \text{corrected ml } KMnO_4}$$

Note: A 1.5 gram sample of sodium oxalate (if the Bureau of Standards oxalate sample has a purity of 99.95 percent) requires 44.75 ml of 0.5N $KMnO_4$.

[1]Due to decomposition caused by contamination, all glassware and equipment for purposes of testing shall be properly cleaned and passivated prior to use.

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4.5.2.2 Concentration Determination - Accurately weigh by difference 6 to 8 drops (approximately 0.4 gram) of hydrogen peroxide sample into a clean, glass stoppered 125-ml Erlenmeyer flask, being careful not to allow the H_2O_2 to wet the ground glass joint. Add 35 ml of 1:1 sulfuric acid. Titrate with the standardized $KMnO_4$ solution until a pink color persists for 30 seconds. Utilize temperature correction factor if the temperature of the $KMnO_4$ is different than 25°C. Determine the reagent blank by titrating 35ml of 1:1 sulfuric acid to the same end point. Correct the milliliters of $KMnO_4$ used in the sample titration by subtracting the blank titration. Determine the concentration of H_2O_2 by averaging the results of a minimum of three titrations. The three results should fall within a range of 0.5 percent.

Calculation:

$$\text{Percent } H_2O_2 = \frac{\text{Ml } KMnO_4 \text{ (corrected)} \times \text{normality } KMnO_4 \times 1.701}{\text{Weight } H_2O_2 \text{ sample}}$$

4.5.3 Determination of Tin content.

Reagents:

2N Sodium hydroxide
35% Hydrochloric acid
Ammonium chloride gelatin solution - dilute 12 ml of a 1% gelatin solution to one liter with saturated aqueous ammonium chloride solution.

Stock tin solution - dissolve 0500 g pure tin in the minimum quantity of 35% HCl then make up to 200 ml with 17% HCl.

Standard tin solution: - Dilute 10 ml of stock tin solution to 100 ml with distilled water. This solution contains 0.25 mg of Sn per ml.

Note; The stock tin solution will keep indefinitely, but the standard solution should be made up fresh on the day it is to be used.

PROCEDURE:

Measure 50 ml of hydrogen peroxide and pour into a 500 ml tall form beaker. Add an equal volume of distilled water and 5 ml of 2N NaOH. Cover with a watch glass and warm gently until the hydrogen peroxide begins to decompose vigorously. When decomposition has nearly ceased, bring to boiling and reduce the volume to approximately 10 ml.

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Run 7.5 ml of 35% HCl from a burette into a 50 ml volumetric flask and measure 30 ml of ammonium chloride gelatin solution in a graduated cylinder. When the decomposed hydrogen peroxide solution has been reduced to a volume of about 10 ml, cool and pour into the HCl in the 50 ml volumetric flask, washing the beaker into the flask with the ammonium chloride gelatin solution. Make up to volume with distilled water.

Transfer to a polarographic cell and bubble nitrogen for 15 minutes.

Measure on the polarograph from -0.3 volts to -0.9 volts versus a saturated calomel electrode. Plot a polarogram.

STANDARDS:

Make up standards containing 0,2,5,7, and 10 ml portions of the standard tin solution in distilled water and add the reagents as above. Polarograph these standards exactly as above.

Note: Unidentified impurities in hydrogen peroxide may result in an ambiguous polarogram. In such case the procedure should be modified as follows:

Measure 50 ml of hydrogen peroxide and pour into a 500 ml tall form beaker. Add an equal volume of distilled water and 5 ml of 2N NaOH. Cover with a watch glass and warm gently until the hydrogen peroxide begins to decompose vigorously. When decomposition has nearly ceased, bring to boiling, evaporate to dryness and subject the residue for two hours to a temperature of 325 to 350°C. Cool, add 5 to 10 ml of water and 7.5 ml of 35% HCl. Allow sufficient time for the dissolution of tin. Transfer to a 50 ml volumetric flask, washing the beaker into the flask with 30 ml of ammonium chloride gelatin solution. Make up to volume with distilled water. Transfer to a polarographic cell and proceed as described above.

4.5.4 Determination of phosphate content - Evaporate 10 ml of hydrogen peroxide to dryness on the steam bath. Subject the dry residue to 325-350°C for two hours. Cool, take-up the residue in 5 ml of approximately IN sulfuric acid, and digest on a steam bath for at least 30 minutes. Cool and transfer solution into a 50 ml volumetric flask, rinsing with distilled water. Add 5 ml of a 5% ammonium molybdate solution (in IN sulfuric acid) and 2 ml of 0.2 percent p-methyl aminophenol sulfate solution containing 20 percent sodium bisulfite. Make up to 50 ml with distilled water, and allow to stand at room temperature for one half hour. Measure the optical density of the solution and determine Po_4 concentrations under identical conditions.

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4.5.5 Determination of nitrate content - Add 10 ml of distilled water and 1 ml of a one percent sodium carbonate solution to 5 ml of hydrogen peroxide. Evaporate to dryness on a steam bath. Wash down the inside of the container with about 5ml of water and again evaporate to dryness. Repeat the water wash and re-evaporate. Add 2 ml of phenol disulfonic acid and heat on the steam bath for 15 minutes. Cool, dilute to 50 ml and make alkaline with concentrated ammonium hydroxide. Dilute to 100 ml. Determine nitrate content by colorimetric comparison with standard sodium nitrate solutions.

Note: In order to prevent any orange or brown tints in the final color development, it is essential that H_2O_2 be absent during the heating step with phenol disulfonic acid.

4.5.6 Determination of chloride content - Dilute 50 ml of concentrated hydrogen peroxide with 48 ml of distilled water and add 1 ml of concentrated nitric acid (sp.gr.1.4) and 1 ml of 1 percent silver nitrate reagent. Any turbidity should not exceed that produced by 0.05 mg of chloride ion in an equal volume of solution containing the quantities of reagents used in the test.

4.5.7 Determination of sulfate content - Evaporate 25 ml to dryness on the steam bath. Take up in 10 ml of water and 1 ml of dilute hydrochloric acid (1 / 19). Transfer to a comparison tube and add 1 ml of 10% of barium chloride reagent solution. Dilute to 50 ml. Any turbidity should not exceed that produced by 0.25 mg sulfate (SO_4) in an equal volume of solution containing the quantities of reagents used in the test.

4.5.8 Evaporation residue - Place some pieces of clean platinum (having a minimum surface area equivalent to two 100 ml evaporating dishes) into a clean 2,000 ml pyrex beaker containing 500 ml of distilled water. Cover with a clean watch glass and place on a hot plate. Add 300 ml sample of the peroxide to be tested in not larger than 50 ml increments. Allow the major portion of each increment to decompose before adding any more. If the solution is maintained at its boiling point, the decomposition operation requires 2 to 4 hours. When the peroxide is almost decomposed, transfer the solution to a clean 1,000 ml pyrex boiling flask, covered with a clean ribbed watch glass. Place a small piece of clean platinum in the boiling flask to prevent concentrating the undecomposed peroxide and boil the solution until it is condensed to 25 to 50 ml volume. Transfer the remaining solution to a 100 ml platinum evaporating dish which has been cleaned, ignited, cooled, and weighed accurately. Place the evaporating dish in an oven at 105° to 110°C for a minimum of 1 hour after all the solution is evaporated (overnight). While decomposing and condensing the peroxide solution, place 500 ml of distilled water into another clean pyrex 1,000 ml boiling flask and in the same manner as specified herein,

they are not electrical rejects.

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evaporate all but 25 to 50 ml, transfer to another weighed platinum dish, and place in the oven. Remove the dry evaporating dishes from the oven, cool in a desiccator, and accurately reweigh each.

Calculation: Subtract the weight gain of the distilled water residue blank from the weight gain of the peroxide residue sample.

$$\text{Milligrams per liter evaporation residue} = \frac{\text{Milligrams (corrected) peroxide residue}}{0.3}$$

4.5.9 - Stability (percent loss of active oxygen without added contaminant) - Place approximately 80 ml of the peroxide to be tested into each of at least three 100 ml pyrex volumetric flasks which have been specially prepared (see notes 1 to 5). Cover the tops of the flasks immediately with a clean piece of pure aluminum foil so that the aluminum is concave across the mouth of the flask (the concave shape tends to prevent condensed droplets from escaping the flask). Weigh the flasks with contents accurately to plus or minus 0.01 gram. Place the flasks in a boiling water or steam bath so that the contents are maintained at 100° ± 1°C. The steam must be sealed around the outside of the necks of the flasks so that it cannot enter the tops of the flasks. A minimum of 2 1/2 inches of the necks of the flasks must extend above the steam seal so that sufficient condensing surface is available to the peroxide. After 24 hours at 100°C remove the flasks, allow to cool, and reweigh to plus or minus 0.01 gram. Determine the percent active oxygen loss by averaging the results of the individual flasks.

Calculation:

Percent active oxygen loss =

$$\frac{W_1 - W_2}{CW_1 \times .470} \times 100$$

W_1 = initial net weight

W_2 = final net weight

C = weight fraction H_2O_2 (concentration/100)

Notes: The special preparation for test flasks is as follows:

(1) Dry new 100 ml pyrex volumetric flasks and obtain tare weights (to 0.01 gram) with aluminum covers on.

(2) Fill the flasks with concentrated certified pure (c.p.) nitric acid and maintain at 100°C. for 24 hours. Cover the tops of the flasks with small beakers, as the aluminum covers are attacked by the acid.

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(3) Remove the acid and rinse the flasks with distilled water. Follow immediately with a peroxide rinse.

(4) Screen the flasks by performing the stability test using peroxide from the same container in each flask. Reject flasks which give inconsistent results.

(5) The flasks are then to be used for no other purpose but to contain peroxide. Even distilled water cannot be allowed to dry in the flasks, but must be rinsed out immediately with peroxide.

4.5.10 Acidity, pH - The apparent pH of hydrogen peroxide is determined by means of a commercial pH meter using a standard glass electrode and a saturated calomel electrode as the reference electrode.

4.5.11 Surface Tension - The surface tension of hydrogen peroxide is determined by means of the "capillary rise method" using a capillary pyrex tube of uniform bore, approximately 0.1 mm radius, thoroughly cleaned and successively passivated with concentrated c.p. nitric acid and 90 percent hydrogen peroxide.

The surface tension Yt in dynes per centimeter is given by the expression

$$Yt = 1/2\ r\ h\ d\ g$$

where

t is the temperature

r the radius of the capillary, in cm

h the height of the hydrogen peroxide column in tne capillary

d the density of hydrogen peroxide at temperature t

g the acceleration due to gravity in cgs units

5. PREPARATION FOR DELIVERY

5.1 Hydrogen peroxide shall be furnished in accordance with one of the following procedures: Contractors shall ensure that drums, tank cars or other containers are thoroughly cleaned prior to filling.

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5.1.1 Drums - The material shall be furnished in aluminum drums with vented closure, in accordance with section 73.266 (a) (2) of the Interstate Commerce Commission regulations for shipping and marking hydrogen peroxide solution, conforming to Specification 42D of the Regulations for Transportation of Explosives and Other Dangerous Articles (see Code of Federal Regulations 49 CFR 71-78). Closure shall be wire sealed to prevent removal in transit. (For shipments other than carload or truck load lots loaded by consignor and unloaded by consignee, the drums shall be of a design and venting arrangement approved by the Bureau of Explosives.) Unless otherwise specified by the bureau or agency concerned, aluminum in contact with the hydrogen peroxide shall be of 99.6 percent purity (.04 copper, maximum, covered by Specification MIL-A-799).

5.1.2 Portable Tanks - Portable tanks complying with Parts 71-78 of Transportation of Explosives and Other Dangerous Articles will be acceptable provided that unless otherwise specified by the bureau or agency concerned aluminum in contact with hydrogen peroxide shall be of 99.6 percent purity (.04 copper, maximum, covered by Specification MIL-A-799).

5.1.3 Tank cars - Hydrogen peroxide solutions in water (70 and 90 percent hydrogen peroxide by weight) may be shipped in tank cars, portable tanks, or tank motor vehicles subject to Parts 71-78 of Transportation of Explosives and Other Dangerous Articles. Tank cars and tank motor vehicles shall be of design and venting arrangement approved by the Bureau of Explosives.

5.2 Marking - In addition to any special marking required in the contract or order, marking for shipment shall be in accordance with Interstate Commerce Commission Regulations and Standard MIL-STD-129.

5.2.1 The following marking data required by Standard MIL-STD-129 not required by Interstate Commerce Commission Regulations shall be stenciled or printed in accordance with Standard MIL-STD-129:

1. Stock number
2. Specification number (latest issue in effect).
3. Hydrogen peroxide concentration (in percent).
4. Name and address of contractor
5. Contract or order number
6. Bill of lading or shipping order number.
7. Domestic or overseas address (as applicable).

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6. NOTES

6.1 Intended Use - The E-stabilized hydrogen peroxide solutions procured under this specification are intended for use as oxidizers in the propulsion systems of chemically powered torpedoes.

6.2 Ordering data - Procurement documents should specify the following:

(a) Title, number, and date of this specification

(b) Grade (see 1.2)

(c) When other than 99.6 percent purity of aluminum is required for drums, tank cars, portable tanks, motor vehicle tanks (see sec. 5).

6.3 WARNING

Concentrated Hydrogen Peroxide should be handled with extreme caution. Instructions concerning the dangers that may be encountered, and methods for its safe handling and use can be found in NAVORD Report 5216, Introduction to Hydrogen Peroxide, and in NAVORD Report 543, Navol Loading Depot.

NOTICE.-- When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.