METRIC

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FEDERAL SPECIFICATION

SULFURIC ACID, ELECTROLYTE (FOR STORAGE BATTERIES)

The General Services Administration has authorized the use of this federal specification by all federal agencies.

1. SCOPE AND CLASSIFICATION

1.1 <u>Scope</u>. This federal specification covers four classes of sulfuric acid for use as an electrolyte in lead-acid storage batteries.

1.2 <u>Classification</u>. Sulfuric acid, electrolyte shall be of the following classes as specified (see 6.2).

Class 1 - Concentrated, minimum 93.2 percent sulfuric acid.

Class 2 - Diluted 49.5 to 50.5 percent sulfuric acid.

Class 3 - Diluted 36.5 to 37.5 percent sulfuric acid.

Class 4 - Diluted 28.3 to 29.5 percent sulfuric acid.

1.3 <u>Unit of issue</u>. The unit of issue and number of units shall be as specified in the acquisition order (see 6.2).

2. APPLICABLE DOCUMENTS

2.1 <u>Government publications</u>. The issues of the following documents, in effect on the date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

Beneficial comments, recommendations, additions, deletions, clarifications, etc. and any data that may improve this document should be sent to: Defense Supply Center Richmond, ATTN: DSCR-VBD, Richmond, VA 23297-5610.

AMSC N/A

FSC 6810

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

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Federal Standard:

FED-STD-313 - Material Safety Data, Transportation Data, and Disposal Data for Hazardous Materials Furnished to the Government

(Activities outside the Federal Government may obtain copies of the federal specifications, standards and commercial item descriptions as specified in the General Information section of the Index of Federal Specifications, Standards and Commercial Item Descriptions. The Index is for sale on a subscription basis form the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.)

(Single copies of this specification, and other federal specifications and commercial item descriptions required by activities outside the Federal Government for bidding purposes are available without charge from the General Services Administration, Federal Supply Service Bureau, Specification Section, Suite 8100, 470 L'Enfant Plaza, SW, Washington, DC 20407.)

(Federal Government activities may obtain copies of federal standardization documents and the Index of Federal Specifications, Standards, and Commercial Item Descriptions from established distribution points in their agencies.)

Military Specification:

MIL-STD-129 - Marking for Shipment and Storage

(Copies of military specification and standards required by contractors in connection with specification procurement functions are obtained from the Standardization Documents Order Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.)

Code of Federal Regulations (CFR):

Title 29, Part 1910.1200 -	Hazardous Communication
Title 49, Parts 100 to 199 -	Department of Transportations Hazardous Materials
	Regulations

(The Code of Federal Regulations (CFR) and the Federal Register (FR) are for sale on a subscription basis for the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. When indicated, reprints of certain regulations may be obtained from the federal agency responsible for issuance thereof.)

Federal Acquisition Regulations (FAR)

(The Federal Acquisition Regulations (FAR) is for sale from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.)

2.2 <u>Other publications</u>. The following documents form a part of this specification to the extent specified herein. Unless a specific issue is identified, the issue in effect of the date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials (ASTM) Standards:

ASTM D 1193	-	Standard Specification for Reagent Water (DoD adopted)
ASTM D 4169	-	Standard Practice for Performance Testing of Shipping
		Containers and Systems (DoD adopted)
ASTM D 4919	-	Standard Specification for Testing of Hazardous Materials Packaging (DoD adopted)

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

International Civil Aviation Organization

Technical Instructions for the Safe Transport of Dangerous Goods by Air

(Application for copies should be addressed to the International Civil Aviation Organization, 100 Sherbrooke Street West, Suite 400, Montreal, Quebec, Canada H3A 2R2.)

International Maritime Organization

International Maritime Dangerous Goods Code

(Application for copies should be addressed to the International Maritime Organization, 101-104 Piccadilly, London, WIV OAE, England.)

3. REQUIREMENTS

3.1 <u>Material</u>. Sulfuric acid, electrolyte shall be non-fuming and free from sediment when tested as specified in 4.3.2.

3.2 <u>Color</u>. Sulfuric acid, electrolyte shall have a color no darker than that of a standard color solution when tested as specified in 4.3.3.

3.3 <u>Acid content and specific gravity</u>. The blend of sulfuric acid, electrolyte shall conform to the characteristics given in table I when tested for acid content as specified in 4.3.4 and specific gravity as specified in 4.3.5.

Class	Acid content, % by Weight		Specific Gravity at 15.6 °C/15.6 °C (60 °F/60 °F)	
	Minimum	Maximum	Minimum	Maximum
1	93.2		1.8354	
2	49.5	50.5	1.3945	1.4042
3	36.5	37.5	1.2767	1.2853
4	28.3	29.5	1.2085	1.2185

TABLEI	Acid	content and	specific	oravity
1 1 1 1 1 1	Titlu	content and	specific	<u>Slavity</u> .

3.4 <u>Impurities</u>. Sulfuric acid, electrolyte shall conform to the maximum impurities characteristics of table II when tested as specified therein.

Impurity	% by weight				Test
	<i>y c</i>				paragraph
	Class 1	Class 2	Class 3	Class 4	
Organic matter	*	*	*	*	4.3.6
Platinum	*	*	*	*	4.3.7
Fixed residue	0.03	0.016	0.012	0.009	4.3.8
Sulfurous acid	0.004	0.0022	0.0016	0.0013	4.3.9
Iron	0.005	0.0027	0.0020	0.0016	4.3.11
Copper	0.005	0.0027	0.0020	0.0016	4.3.12**
Zinc	0.004	0.0022	0.0016	0.0013	4.3.13
Arsenic	0.0001	0.00005	0.00004	0.00003	4.3.14
Antimony	0.0001	0.00005	0.00004	0.00003	4.3.15**
Selenium	0.002	0.0011	0.0008	0.0006	4.3.16**
Nickel	0.0001	0.00005	0.00004	0.00003	4.3.17
Manganese	0.00002	0.000011	0.000008	0.000006	4.3.18
Nitrate	0.0005	0.00027	0.00020	0.00016	4.3.19
Ammonium	0.001	0.0005	0.0004	0.0003	4.3.20
Chloride	0.001	0.0005	0.0004	0.0003	4.3.21

TABLE II. Maximum impurities.

* To pass test.

** See 4.3.10 for preliminary test.

3.5 <u>Recovered materials</u>. The offeror/contractor is encouraged to use recovered materials to the maximum extent practicable, in accordance with paragraph 23.403 of the Federal Acquisition Regulations (FAR).

4. QUALITY ASSURANCE PROVISIONS

4.1 <u>Responsibility for inspection</u>. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements (examinations and tests) as specified herein. Except as otherwise specified in the contract or purchase order, the contractor 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 this specification where such inspections are deemed necessary to ensure supplies and services conform to prescribed requirements.

4.1.1 <u>Responsibility for compliance</u>. All items shall meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of ensuring that all products or supplies submitted to the government for acceptance comply with all requirements of the contract. Sampling inspection, as part of the manufacturing operations, is an acceptable practice to ascertain conformance to requirements, however, this does not authorize submission of known defective material, either indicated or actual, nor does it commit the government to accept defective material.

4.1.2 <u>Contractor assurance of compliance</u>. The contractor's quality program or detailed inspection system shall provide assurance of compliance of all characteristics with the applicable drawing and specification requirements using, as a minimum, the conformance criteria specified herein.

4.1.3 <u>Alternative inspection provisions</u>. Alternate inspection procedures, methods or equipment, such as statistical process control, tool control, and other types of sampling procedures may be used by the contractor when they provide, as a minimum, the level of quality assurance required by the inspection provisions specified herein. Prior to applying such alternative procedures, methods, or equipment, the contractor shall describe them in a written proposal submitted to the government for evaluation and approval (see 6.6). When required, the contractor shall demonstrate that the effectiveness of each proposed alternative provided is equal to or better than the quality assurance provisions specified herein. In cases of dispute as to whether the contractor's proposed alternative provides equal quality assurance, the provisions of this specification shall apply. All approved alternative inspection provisions shall be specifically incorporated into the contractor's quality program or detailed inspection system, as applicable.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the sulfuric acid, electrolyte of one class offered for acceptance at one time which has been produced by one manufacturer, at one plant, during one shift, for the same materials, and under essentially the same manufacturing conditions provided the operation is continuous. In the event the process is a batch operation, each batch shall constitute a lot (see 6.4).

4.2.2 <u>Sampling for sulfuric acid, electrolyte test</u>. See 6.5 for sampling and testing precautions. Sampling shall be conducted in accordance with table III. A representative specimen of approximately 1 liter shall be removed from each sample container and placed in a suitable clean, dry container labeled to identify the lot and container from which it was taken.

Number of containers in batch or lot	Number of sample containers
2 to 50	2
51 to 500	3
501 to 35,000	5
Over 35,000	8

TABLE III. Sampling for sulfuric acid, electrolyte tests.

4.2.3 <u>Inspection procedures for sulfuric acid, electrolyte tests</u>. Each sample specimen taken in 4.2.2 shall be tested as specified in 4.3.5. Failure of any test by any specimen shall be cause for rejection of the lot represented.

4.3 <u>Test methods</u>.

4.3.1 <u>Sulfuric acid, electrolyte tests</u>. See 6.5 for sampling and testing precautions. The water shall be in accordance with ASTM D 1193 and reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections applied where significant. Unless otherwise specified, acid and base reagents shall be concentrated acids and bases. Tests shall be conducted as follows:

4.3.2 <u>Material</u>. Visually examine the specimen for evidence of fuming and for presence of sediment.

4.3.3 <u>Color</u>. Compare the color of 50 ml of the specimen in a Nessler tube with that of 50 ml of a standard contained in a second, matching Nessler tube. Prepare the standard using 0.12 mg of copper, 1 ml of 0.1 N hydrochloric acid, and 50 ml of freshly prepared hydrogen sulfide water. Dilute to 50 ml for class 1 comparison, 120 ml for class 2 comparison, 180 ml for class 3 comparison, or 260 ml for class 4 comparison. Make the comparison vertically through the solution immediately after the preparation of the standard. The matching Nessler tubes should have a capacity of 50 ml and approximately dimensions of 30 cm in length by 2 cm in diameter.

4.3.4 <u>Acid content</u>. By means of a weighing buret, weighing bottle, or Dely tube, transfer an accurately weighed amount of the specimen, equivalent to 0.8 to 1.1 g of 100 percent sulfuric acid, into a porcelain casserole dish containing 100 to 150 ml of carbon-dioxide free water. Add 1 ml of phenolphthalein indicator (1 g in 50 ml ethyl alcohol and 50 ml water) and titrate to a faint permanent pink color with standard 0.5 N sodium hydroxide solution. Record the volume and temperature of 0.5 N sodium hydroxide solution (titer) used. Correct the volume of the titer by adding 0.00035 ml per milliliter of titer for each degree C that the sodium hydroxide solution is below the temperature of standardization. Calculate the percent by weight sulfuric acid as follows:

Percent sulfuric acid = $\frac{4.904AB}{W}$

where: A = Correct milliliter of sodium hydroxide solution used in the titration,

- B = Normality of the sodium hydroxide solution, and
- W = Weight of specimen in grams.

4.3.5 <u>Specific gravity</u>. Determine the specific gravity of the specimen using a suitable precision hydrometer standardized at 15.6 °C (60 °F), referred to water at 15.6 °C (60 °F). Make temperature corrections, when necessary, for deviations from the standard temperature as shown in table IV. When the temperature of the acid is above the standard temperature, add the correction to the observed specific gravity; when the temperature of the acid is below the standard temperature, subtract the correction from the observed specific gravity factor.

Class of	Correction per degree of		
sulfuric acid	temperature deviation		
	°C	°F	
1	0.00097	0.00054	
2	0.00074	0.00041	
3	0.00070	0.00039	
4	0.00065	0.00036	

TABLE IV. Correction for temperature deviation.

4.3.6 <u>Organic matter</u>. Heat 50 ml of the specimen in a clean beaker until the sulfuric acid begins to fume strongly. To pass test, there shall be no perceptible charring; a slight yellow color that appears when a specimen is hot but disappears on cooling should be disregarded.

4.3.7 Platinum. As applicable, evaporate 10 ml of class 1, 24 ml of class 2, 36 ml of class 3, or 52 ml of class 4 specimen to dryness in a small porcelain evaporation dish set on a sand bath, but do not heat the residue unnecessarily. Cool and add 2 ml of agua regia (4 volumes of hydrochloric acid and 1 volume of nitric acid). Cover the dish with a watchglass and digest on a steam bath for 10 minutes. Remove the watchglass and evaporate the solution to five or six drops. (If the solution accidentally evaporates completely, repeat the digestion with agua regia and reevaporate to five or six drops.) Absorb the solution (five or six drops) in a piece of thin asbestos paper preferably not over 0.75 mm thick and about 0.5 cm wide and 3 cm long. (Hold the paper in a pair of crucible tongs and dip half of the paper into the solution. Dry gently over a flame. Repeat the dipping and drying operation until all of the solution has been absorbed into the asbestos paper.) Dry the paper and ignite to redness in a moderate Bunsen flame (3 to 4 inches high with good air mixture). With the asbestos paper held vertically in the Bunsen flame, momentarily shut off the flow of gas and then turn it on again so that the stream of unignited gas plays on the hot asbestos paper. Glowing of the paper indicates the presence of platinum or one of the platinum metals other than osmium or ruthenium. (Note: The glow should persist; however, if it dies out, it may be brought back by reigniting the paper, although this cannot be repeated indefinitely.) The presence of platinum shall not be indicated.

4.3.8 <u>Fixed residue</u>. As applicable, measure 10 ml of class 1, 24 ml of class 2, 36 ml of class 3, or 52 ml of class 4 specimen into a platinum dish, evaporate to dryness, and ignite at a dull red heat for five minutes. Cool in a desiccator and weigh the residue (save the residue for iron and copper determinations). Calculate the percent by weight fixed residue as follows:

Percent fixed residue = $\frac{100A}{BW}$

Where A = Weight of residue in grams,

B = Milliliters of specimen, and

W = Specific gravity of the specimen determined in 4.3.5.

4.3.9 <u>Sulfurous acid</u>. As applicable, dilute 20 ml of class 1, 49 ml of class 2, or 72 ml of class 3 specimen to 80 ml with water; in the case of class 4 specimen, evaporate 104 ml to approximately 75 ml and dilute to 80 ml with water. Cool the 80 ml of solution to 25 °C and titrate with 0.1 N potassium permanganate solution to a faint but permanent pink color. No more than 0.46 ml of 0.1 N permanganate solution shall be required. Save the titrated solution for use in 4.3.10.

4.3.10 <u>Preliminary test for antimony, copper, and selenium</u>. Dilute the titrated solution from 4.3.9 with water to 150 ml and, if necessary, filter off any precipitate. Pass hydrogen sulfide through the solution for 5 minutes, let stand for 10 minutes, and filter without washing through a 9 cm quantitative filter paper to close texture (save the filtrate for 4.3.17). Unfold the paper and examine it closely, especially near the center, in daylight or equivalent artificial light. Absence of a black or brown color indicates that less than the permitted amount of copper is present and the determination of copper is to be omitted. If the paper, in the absence of a black or brown color, shows neither a light yellow nor an orange color, antimony and selenium are less than the permitted amounts and the determination of these impurities is to be omitted. (Note: If sulfur precipitates during the hydrogen sulfide treatment, the preliminary test for antimony may be meaningless. The precipitation of sulfur will not obscure copper or selenium, if present.)

4.3.11 <u>Iron</u>. (If the percent fixed residue determined in 4.3.8 does not exceed 0.0070 for class 1, 0.0038 for class 2, 0.0028 for class 3, or 0.0022 for class 4, the determination of iron is to be omitted.) Add 1 ml of hydrochloric acid to the residue retained in 4.3.8, warm on a steam bath to dissolve the residue, and dilute to 10 ml in a volumetric flask (solution A). Dilute a 1 ml aliquot of solution A to 100 ml in a volumetric flask (solution B). (Save the remaining 9 ml of solution A for the copper determination.) To a 20 ml aliquot of solution B, add 1 ml of hydrochloric acid, 30 to 50 mg of ammonium persulfate, and 3 ml of 30 percent ammonium thiocyanate solution. The color developed shall not exceed that developed in a standard containing 0.018 mg of iron and the same quantities of reagents made up to an equal volume.

4.3.12 <u>Copper</u>. (If the percent fixed residue determination in 4.3.8 does not exceed 0.0060 for class 1, 0.0032 for class 2, 0.0024 for class 3, or 0.0019 for class 4, or if the preliminary test for copper in 4.3.10 does not indicate the presence of copper, the determination of copper is to be omitted.) Neutralize the remaining 9 ml of solution A from 4.3.11 with ammonium hydroxide and add 4 ml in excess. Heat sufficiently to coagulate any precipitate, filter into a Nessler tube,

and dilute to about 20 ml. The blue color shall not be deeper than that produced by 0.83 mg of copper in an equal volume of solution containing 4 ml of ammonium hydroxide.

4.3.13 <u>Zinc</u>. (If the percent fixed residue does not exceed 0.0060 for class 1, 0.0032 for class 2, 0.0024 for class 3, or 0.0019 for class 4, determination of zinc is to be omitted.) As applicable, dilute 20 ml of class 1, 49 ml of class 2, 72 ml of class 3, or 104 ml of class 4 specimen to approximately 110 ml and neutralize with ammonium hydroxide. Dilute the solution to 200 ml, add 10 ml of 6 N sulfuric acid, and saturate with hydrogen sulfide gas. Filter the solution and boil the filtrate to remove the hydrogen sulfide. Cool the solution and neutralize with ammonium hydroxide using methyl red as the indicator. Pipette 1 ml of 1 N sulfuric acid for each 100 ml of solution into the solution and pass a rapid stream of hydrogen sulfide through the solution for 20 minutes. Collect any precipitate formed on ashless filter paper, wash with water, ignite in a tared crucible, cool to room temperature in a desiccator, and weigh the collected precipitate. Calculate the percentage weight zinc as follows:

Percent Zinc = 100A/BW

where: A = Weight of collected precipitate in grams,

B = Milliliters of specimen, and

W = Specific gravity of the specimen

4.3.14 <u>Arsenic</u>. As applicable, dilute 20 ml of class 1, 49 ml of class 2, 58 ml of class 3, or 78 ml of class 4 specimen to 100 ml in a volumetric flask. As applicable, dilute a 20 ml aliquot of the class 1 or class 2 specimen dilution or a 25 ml aliquot of the class 3 or class 4 specimen dilution to 50 ml and determine arsenic by modified Gutzeit procedure. The stain produced shall not exceed that produced by 0.0075 mg of arsenic. (Note: Also compare the stain with that produced by 0.00075 mg of arsenic in reference 4.3.15.)

4.3.15 <u>Antimony</u>. If arsenic content has been found to be not greater than one-tenth of the permitted limit (see note of 4.3.14), antimony is determined without first removing arsenic; proceed as specified in 4.3.15.1 below. If the arsenic content has been found greater than one-tenth the permitted limit, proceed as specified in 4.3.15.2 below.

4.3.15.1 <u>Antimony - arsenic content is less than or equal to 0.0001 percent.</u> As applicable, evaporate 11 ml of class 1, 27 ml of class 2, 40 ml of class 3, or 57 ml of class 4 specimen to 4 or 5 ml. Transfer to a Gutzeit apparatus, make up to 50 ml with water, and proceed as in the determination of arsenic except that the solution is finally warmed to 60 °C and kept at $60 \circ \pm 3 \circ C$ for 1 hour. Develop the stain by immersing the paper strip (mercuric bromide paper) in 20 percent by volume ammonium hydroxide solution. The stain developed shall not exceed that produced by 0.02 mg of antimony following the same procedure.

4.3.15.2 <u>Antimony - arsenic content is greater than 0.0001 percent.</u> As applicable, evaporate 55 ml of class 1, 110 ml of class 2, 162 ml of class 3, or 225 ml of class 4 specimen to 4 or 5 ml in a 250 ml beaker. Cool to room temperature and cautiously add 10 ml of water containing 0.25 g of sodium sulfite. Mix and evaporate until the solution begins to fume. Cool and add

100 ml of hydrochloric acid. Boil slowly (in the open beaker) until the volume is reduced to 40 or 50 ml keeping the temperature of the liquid bellow 110 °C. (Use boiling tubes or chips to minimize bumping and take care not to let the volume get too small or the temperature too high; otherwise some antimony may be lost.) Transfer the reduced volume quantitatively, with a minimum amount of water, to a 50 ml volumetric flask and make up to volume with water. To a 10 ml aliquot, add 10 ml of water and 3 ml of reagent sulfuric acid. Mix, transfer to a Gutzeit apparatus, dilute to 50 ml and proceed as specified in paragraph 4.3.15.1 above. The stain developed shall not exceed that produced by 0.02 mg of antimony, 3 ml of reagent sulfuric acid, and 4 ml of hydrochloric acid.

4.3.16 <u>Selenium</u>. (If preliminary test for selenium in 4.3.10 was negative, the determination of selenium is to be omitted.) Classes 2, 3 and 4 specimens required concentrating prior to determining selenium; concentrate by evaporating 20 to 30 ml until the heavy fumes of sulfuric acid appear. As applicable, place 3 ml of class 1 or concentrated class 2, 3, or 4 specimen in a test tube. Cool the test tube and contents in an ice bath. Overlay the test tube contents with 10 ml of hydrochloric acid containing approximately 10 mg of sodium sulfite. (Note: Initially, with care, add just enough of the hydrochloric acid to cover the surface of the contents in the test tube, wait until any effervescence ceases, then continue to overlay the remaining portion of the 10 ml of hydrochloric acid.) Observe the zone of contact between the two liquids for 3 minutes from the time the specimen is completely covered by the first addition of hydrochloric acid. The formation of a red to reddish-brown ring denotes the presence of selenium content exceeding the permitted limit. (Note: In the presence of iron or copper, a yellow ring will form in the zone of contact. The red to reddish-brown ring of selenium, if present, will form slightly above and separate from the yellow ring of iron or copper. The red to reddish-brown ring should be viewed transversely against a white background and not be confused with the yellow ring.)

4.3.17 <u>Nickel</u>. Gently boil 50 ml of the filtrate retained from 4.3.10 to remove the excess hydrogen sulfide. Cool to room temperature, add 2 ml of 20 percent tartaric acid solution and 2 ml of water saturated with bromine. Slowly add ammonium hydroxide solution until the bromine color is discharged. Cool the solution to room temperature. Add 2 ml of ammonium hydroxide solution, 2 ml of 1 percent solution of dimethylglyoxime in alcohol, and 1 ml of water saturated with bromine. Any color produced shall not exceed that produced by 0.012 mg of nickel in an equal volume of solution treated in the same manner as the specimen solution filtrate after boiling.

4.3.18 <u>Manganese</u>. As applicable, dilute 20 ml of class 1, 49 ml of class 2, or 72 ml of class 3 specimen to 95 ml with water and add 5 ml of nitric acid; in the case of class 4 specimen, evaporate 104 ml to approximately 90 ml, dilute to 95 ml with water, and add 5 ml of nitric acid. Add 5 ml of 85 percent phosphoric acid. Add 0.5 g of potassium periodate and boil the solution vigorously in a covered beaker for 5 minutes. Cool to room temperature. The color shall be no deeper than that of a standard containing 0.7 ml of 0.001 N potassium permanganate solution diluted to 95 ml with water and treated in the same manner.

4.3.19 Nitrate.

4.3.19.1 <u>Standard nitrate solution</u>. Prepare a stock nitrate solution by dissolving 0.625 g of potassium nitrate in water in a 1 liter volumetric flask and make up to volume. Prepare a standard nitrate solution by pipeting 20 ml of the stock solution into a 1 liter volumetric flask and make up to volume. (The standard solution contains 0.00766 mg of nitrate per milliliter.)

4.3.19.2 <u>Class 1</u>. Pipet 10 ml of water into one of a matched pair of Nessler tubes and add, by pipet, 5 ml of class 1 specimen and 15 ml of nitrate free reagent sulfuric acid. Into the second Nessler tube, pipet 4 ml of water and 6 ml of the standard nitrate solution prepared in 4.3.19.1. Add, by pipet, 20 ml of nitrate free reagent sulfuric acid. To each Nessler tube, add 0.5 ml of a saturated solution of ferrous sulfate and mix. Immediately after mixing, the color or darkening of the solution in the Nessler tube containing the class 1 specimen shall not exceed that of the solution in the Nessler tube containing the standard nitrate solution. (Note: Only the difference in color is to be considered, not the turbidity which may be present due to the precipitation of lead sulfate when the specimen is diluted.)

4.3.19.3 <u>Class 2</u>. Pipet 10 ml of class 2 specimen into one of a matched pair of Nessler tubes and add, by pipet, 10 ml of nitrate free reagent sulfuric acid. Into the second Nessler tube, pipet 5 ml of the standard nitrate solution prepared in 4.3.19.1 and add, by pipet, 15 ml of nitrate free reagent sulfuric acid. To each Nessler tube, add 0.5 ml of a saturated solution of ferrous sulfate and mix. Immediately after mixing, the color or darkening of the solution in the Nessler tube containing the class 2 specimen shall not exceed that of the solution in the Nessler tube containing the standard nitrate solution. See note in 4.3.19.2.

4.3.19.4 <u>Class 3</u>. Pipet 15 ml of class 3 specimen into one of a matched pair of Nessler tubes and add, by pipet, 5 ml of nitrate free reagent sulfuric acid. Into the second Nessler tube, pipet 5 ml of the standard nitrate solution prepared in 4.3.19.1 and add, by pipet, 15 ml of nitrate free reagent sulfuric acid. To each Nessler tube, add 0.5 ml of a saturated solution of ferrous sulfate and mix. Immediately after mixing, the color or darkening of the solution in the Nessler tube containing the class 3 specimen shall not exceed that of the solution in the Nessler tube containing the standard nitrate solution. See note in 4.3.19.2.

4.3.19.5 <u>Class 4</u>. Pipet 20 ml of class 4 specimen into one of a matched pair of Nessler tubes and add, by pipet, 10 ml of nitrate free reagent sulfuric acid. Into the second Nessler tube, pipet 5 ml of water and 5 ml of the standard nitrate solution prepared in 4.3.19.1 and add, by pipet, 10 ml of nitrate free reagent sulfuric acid. To each Nessler tube, add 0.5 ml of a saturated solution of ferrous sulfate and mix. Immediately after mixing, the color or darkening of the solution in the Nessler tube containing the class 4 specimen shall not exceed that of the solution in the Nessler tube containing the standard nitrate solution. See note in 4.3.19.2.

4.3.20 <u>Ammonium</u>. As applicable, dilute 20 ml of class 1, 49 ml of class 2, 58 ml of class 3, or 78 ml of class 4 specimen to 100 ml in a volumetric flask. As applicable, in a Nessler tube, dilute 20 ml of class 1 or class 2 specimen dilution or dilute 25 ml of class 3 or class 4 specimen dilution to 90 ml with water, make alkaline with sodium hydroxide solution, and add 1 ml of Nessler's reagent. The color produced shall not exceed that produced by 0.072 mg of ammonium

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ion (1 ml of a 1 liter solution of 0.22 g of ammonium chloride in water) treated in the same manner.

4.3.21 <u>Chloride</u>. As applicable, dilute 20 ml of class 1, 49 ml of class 2, 36 ml of class 3, or 49 ml of class 4 specimen to 100 ml in a volumetric flask. As applicable, in a Nessler tube, dilute 5 ml of class 1 or class 2 specimen dilution or dilute 10 ml of the class 3 or class 4 specimen dilution to 50 ml with water. Add 1 ml of 1 to 10 nitric acid and 1 ml of 2 percent silver nitrate solution. The turbidity produced shall not exceed that produced by 0.018 mg of chloride ion (1 ml of 1 liter solution of 0.030 g of sodium chloride in water) treated in the same manner.

5. PACKAGING

5.1 <u>General requirements</u>. The packaging requirements shall be as specified herein unless modified by the contract or order (see 6.2). When actual packaging of material 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.

5.2 <u>Packaging</u>. Package and pack in accordance with the contractor's packaging design that shall successfully pass the standard practice for Performance Testing in accordance with ASTM D 4919 and ASTM D 4169. When tested in accordance with ASTM D 4169, Distribution Cycle 18 and Assurance Level I shall be used. Acceptance shall be criterion I (no product damage). Unit pack quantity shall be 1 each.

5.2.1 Additional, unit pack preservation and marking of any hazardous material item shall comply with Performance Oriented Packaging (POP) requirements (including testing) as stated in International Civil Aviation Organization-Technical Instructions for Safe Transportation of Dangerous Goods by Air (ICAO-TDGA), International Maritime Organization-International Maritime Dangerous Goods Code (IMO-IMDG), and 49 CFR, Parts 100 to 199. Unit pack of hazardous material must be certified for air transport on cargo aircraft.

5.2.2 All performance test requirements for the unit pack of the hazardous material (electrolyte) shall be supported by certificates, reports attesting to date and type of testing conducted, and test data results collected from POP testing. Only "overpack containers" used to consolidate certified pack containers for shipping purposes shall be exempt from POP testing requirements, but shall comply with all markings and labeling requirements previously stated and shall indicate that the inside containers have been certified, along with the UN markings on the unit pack.

5.3 <u>POP</u>. The contractor shall be a POP self-certified registered with the Department of Transportation (DOT), or responsible for assuring an approved third party source providing POP testing services is in compliance with requirements and registered with DOT (e.g. symbol used to certify compliance with POP requirements must be one which is properly registered with DOT). The contractor's signed certification that the packaged configuration meets 49 CFR, ICAO, and IMDG requirements shall be incorporated into the DD Form 250, Material Inspection and Receiving Report, or other related acceptance document if the DD Form 250 is not used.

5.4 <u>Certificates and reports</u>. All certificates and reports shall be available for inspection by authorized Government representatives for a period of three years after contract completion, and one copy to the design activity and the Procuring Contracting Officer not later than the start of presentation of items to the accepting activity/Contracting Officer.

5.5 <u>Palletization</u>. Palletization shall be used where containers do not require skids and where quantities per destination exceed a total of 115 kilograms (250 pounds).

5.6 <u>Markings</u>. All unit pack and overpacks shall be marked and labeled in accordance with MIL-STD-129, Title 49 CFR, Parts 100-199, ICAO-TDGA, and IMO-IMDG.

5.6.1 A Material Safety Data Sheets (MSDS) shall be prepared in accordance with FED-STD-313 and meeting the requirements of 29 CFR 1910.1200. A MSDS shall be included with each unit package containing a hazardous material.

5.6.2 Where appropriate, shelf-life markings shall be applied in accordance with MIL-STD-129.

6. NOTES

INFORMATION FOR GUIDANCE ONLY. (This section contains information of a general or explanatory nature that is helpful, but is not mandatory.)

6.1 <u>Intended use</u>. Sulfuric acid, electrolyte is intended for use in lead storage batteries. Class 1 sulfuric acid must be diluted to the applicable density specified for class 2, 3, or 4 before use; class 1 sulfuric acid, electrolyte should never be used undiluted in lead storage batteries.

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

- a. Title, number, and date of this specification.
- b. Class of sulfuric acid required (see 1.2).
- c. Unit of issue and number of units required (see 1.3).
- d. Packaging requirements (see 5.1).

6.3 <u>MSDS</u>. Contracting officers will identify those activities requiring copies of completed MSDS prepared in accordance with FED-STD-313 and meeting the requirements of 29 CFR 1910.1200. The pertinent Government mailing addresses for submission of the data are listed in FED-STD-313; and 29 CFR 1910.1200 requires that the MSDS for each hazardous chemical used in an operation must be readily available to personnel using the material. Contracting officers will identify the activities requiring copies of the MSDS.

6.4 <u>Batch</u>. A batch is defined as that quantity of material that has been manufactured by some unit chemical process or subjected to some physical mixing operation intended to make the final product substantially uniform.

6.5 <u>Sampling and testing precautions</u>. This specification requires inspection of chemical material that is potentially hazardous to personnel. This specification does not purport to address all of the safety problems associated with its use. Sulfuric acid is very corrosive and dangerous when improperly handled. Contact with body results in rapid destruction of tissues and severe burns. Adequate protection against any contact should be provided for all parts of the body. Although the acid itself is not flammable, it may cause ignition by contact with combustible materials; a highly flammable gas (hydrogen) is generated by the action of the acid on most metals. Concentrated sulfuric acid (class I) should not be stored in lead containers. Air pressure should never be used to empty carboys. It is the responsibility of the user of this specification to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

6.6 <u>Submission of alternate inspection provisions</u>. Proposed alternative inspection provisions should be submitted by the contractor to the procuring contracting officer for evaluation and approval by the technical activity responsible for preparation of this specification.

6.7 <u>Significant places</u>. For purposes of determining conformance with this specification, an observed or calculated value should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value in accordance with the rounding-off method of ASTM E 29.

6.8 <u>Part or identification number (PIN)</u>. The following PIN procedure is for government purposes and does not constitute a requirement for the contractor.



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6.9 Subject term (key word) listing.

Acid content Aqua regia Ammonium Ammonium hydroxide Antimony Arsenic Asbestos paper Battery acid Chloride Contamination Copper Fixed residue Hydrogen sulfide Hydrochloric acid Iron Leakage Material Safety Data Sheets Manganese Mercuric bromide paper Nickel Nitrate Nitric acid Organic matter Platinum Selenium Shock resistance Specific gravity Sulfurous acid Zinc

MILITARY INTERESTS:

Custodians: Army - EA Navy - SH Air Force - 68

Reviewers: Army - AT, MD, MI Navy - MC, SA, YD

CIVIL AGENCY COORDINATING ACTIVITY:

GSA - 7FXE

Preparing activity: DLA - GS3

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