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April 14, 1965 SUPERSEDING Fed. Spec. O-S-801a August 8, 1956

FEDERAL SPECIFICATION

SULFURIC ACID, ELECTROLYTE; FOR STORAGE BATTERIES

This specification was approved by the Commissioner, Federal Supply Serice, General Services Administration, for the use of all Federal agencies.

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers sulfuric acid and electrolytic solutions for use in storage batteries.

1.2 Classification.

1.2.1 *Classes.* The sulfuric acid and electrolytic solutions shall be of the following classes as specified (see 6.2):

- Class 1—Concentrated sulfuric acid; minimum specific gravity 1.8354 at 60°F/ 60°F.
- Class 2—Dilute sulfuric acid; specific gravity 1.3945 to 1.4042 at 60°F/60°F.
- Class 3-Dilute sulfuric acid; specific gravity 1.2767 to 1.2853 at 60°F/60°F.
- Class 4—Dilute sulfuric acid, specific gravity 1.2085 to 1.2185 at 60°F/60°F.

2. APPLICABLE SPECIFICATIONS, STANDARDS, AND OTHER PUBLICA-TIONS

2.1 Specifications and standards. The following specifications and standards, of

the issues in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

Federal Standards:

Fed. Std. No. 102—Preservation, Packaging, and Packing Levels.

Fed. Std. No. 123-Marking for Domestic Shipment (Civilian Agencies).

(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards, and Handbooks as outlined under General Information in the Index of Federal Specifications and Standards at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

(Single copies of this specification and other product specifications required by activities outside the Federal Government for bidding purposes are available without charge at the General Services Administration Regional Offices in Boston, New York, Washington, D.C., Atlanta, Chicago, Kansas City, Mo., Dallas, Denver, San Francisco, Los Angeles, and Seattle, Wash.

(Federal Government activities may obtain copies

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of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

Military Specifications:

- MIL-S-207—Sulfuric Acid, Electrolyte: Packaging, Packing, and Marking for Shipment and Storage of.
- MIL-D-40030-Drums, Plastic, Molded Polyethylene.

Military Standards:

- MIL-STD-105-Sampling Procedures and Tables for Inspection by Attributes.
- MIL-STD-129-Marking for Shipment and Storage.

(Copies of specifications, standards, drawings, and publications required by suppliers 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 issue in effect on date of invitation for bids or request for proposal shall apply.

Code of Federal Regulations:

49 CFR 71–90—Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles.

(The Interstate Commerce Commission regulations are a part of the Code of Federal Regulations (Revised 1956) available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402. Orders for the above publication should cite "49 CFR 71-90 (Rev. 1956).")

American Society for Testing and Materials (ASTM) Standard: D1193-Reagent Water.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa. 19103.)

3. REQUIREMENTS

3.1 Material. The sulfuric acid shall be non-fuming and free from sediment when examined as specified in 4.6.1.

3.2 Color. The concentrated acid or diluted acid solutions shall be preferably colorless. In no case shall the color be darker than that of a standard color solution when tested as specified in 4.6.2.

3.3 Strength of acid. The sulfuric acid content, by weight, shall be as follows, when tested as specified in 4.6.3.

Class 1—no less than 93.2 percent, Class 2—49.5 to 50.5 percent, Class 3—36.5 to 37.5 percent, and Class 4—28.3 to 29.5 percent.

3.4 Specific gravity. The specific gravity at $60^{\circ}F/60^{\circ}F$ shall be as follows, when tested as specified in 4.6.4 (see 6.3):

Class 1—minimum of 1.8354, Class 2—1.3945 to 1.4042, Class 3—1.2767 to 1.2853, and Class 4—1.2085 to 1.2185.

3.5 Impurities. The maximum limit of impurities in the sulfuric acid or electrolytic solutions shall be as shown in table 1 when tested as specified therein.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection require-

ments as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to 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 that supplies and services conform to prescribed requirements.

TA	BLE	I.— <i>Limit</i>	of	impurities
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Impurity	as	Class 1	Class 2	Class 3	Class 4	test paragraph
Organic matter		*	*	*	*	4.6.5.1
Platinum	Pt	*	*	*	*	4.6.5.2
Fixed residue	-	0.03	0.016	0.012	0.009	4.6.5.3
Sulfurous acid	so,	.004	.0022	.0016	.0013	4.6.5.4
Iron	Fe	.005	.0027	.0020	.0016	4.6.5.6
Copper	Cu	.005	.0027	.0020	.0016	4.6.5.7**
Zinc	Zn	.004	.0022	.0016	.0013	4.6.5.8
Arsenic	As	.0001	.00005	.00004	.00003	4.6.5.9
Antimony	Sb	.0001	.00005	.00004	.00003	4.6.5.10**
Selenium	Se	.002	.0011	.0008	.0006	4.6.5.11**
Nickel	Ni	.0001	.00005	.00004	.00003	4.6.5.12
Manganese	Mn	.00002	.000011	.000008	.000006	4.6.5.13
Nitrates	NO.	.0005	.00027	.00020	.00016	4.6.5.14
Ammonium	NH 4	.001	.0005	.0004	.0003	4.6.5.15
Chloride	Cl	.001	.0005	.0004	.0003	4.6.5.16
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* To pass test.

** See 4.6.5.5 for preliminary test.

4.2 Special provisions.

4.2.1 Alternative inspection. The supplier may utilize any alternative inspection procedure which will provide equal or better assurance of quality by submitting a written proposal with justification and obtaining written approval from the Government prior to instituting the procedure. In case of dispute, the procedures of this specification will govern.

4.2.2 Objective evidence. The supplier shall provide objective evidence acceptable to the contracting officer that the requirements of section 5 for which specific inspection has not been provided in this specification have been satisfied.

4.3 Lotting. A lot shall consist of the sulfuric acid, electrolyte of the same class offered for acceptance at one time, which has been produced by one manufacturer under essentially the same manufacturing conditions and with no change in materials provided the operation is continuous. In the event that the process is a batch operation, each batch shall constitute a lot (see 6.4).

4.4 Sampling.

4.4.1 For examination. Sampling shall be conducted in accordance with MIL-STD-105.

4.4.2 For destructive tests. Sampling

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shall be conducted in accordance with MIL-STD-105 using level S-1. A specimen shall be removed from each container in the sample and placed in a suitable, clean, dry container labeled to identify the lot and the container from which it was taken.

4.5 Inspection provisions.

4.5.1 For examination. The levels A and B preparation for delivery shall be examined in accordance with the classification of defects and MIL-STD-105.

4.5.2 For destructive tests. Samples shall be tested in accordance with 4.6 and MIL-STD-105 using an acceptance number of zero.

4.5.3 Classification of defects.

4.5.3.1 Preparation for delivery (section 5).

Categories	Defects			
Critical:	None defined			
Major:	AQL 1.0 percent defective			
101	Unit container not type or class specified			
102	Unit container closure incorrect or loose			
103	Net contents per unit container less than specified or indicated quan- tity			
104	Shipping container not as specified			
105	Closure of intermediate or shipping container not as specified			
106	Unit or shipping container dam- aged			
107	Marking or labeling of unit or ship- ping container incorrect, illegible, or incomplete			

4.6 Tests. Distilled water or water conforming to ASTM D1193 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:

4.6.1 *Material*. Examine the specimen visualy for compliance with 3.1.

4.6.2 Color. Compare the color of 50 milliliters (ml.) of the acid 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 milligrams (mg.) of copper, 1 ml. of 0.1N hydrochloric acid, 5 ml. of freshly prepared hydrogen sulfide water and 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 solutions immediately after the preparation of the standard. The matching Nessler tubes shall have a capacity of 50 ml. and approximate dimensions of 30 centimeters (cm.) in length by 2 cm. in diameter.

4.6.3 Strength of acid. By means of a weighing buret, weighing bottle, or Dely tube, transfer an accurately weighed amount of the acid, equivalent to 0.8 to 1.1 grams (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. per 100 ml.-50 ml. ethyl alcohol and 50 ml. water) and titrate to a faint permanent pink color with standard 0.5N sodium hydroxide solution. Record the volume and temperature of 0.5N sodium hydroxide solution (titer) used. Correct the volume of the titer by adding 0.00035 ml. per ml. of titer for each degree C. that the sodium hydroxide solution is below the temperature of standardization or by subtracting 0.00035 ml. per ml. of titer for each degree C. that

Calculate

the sodium hydroxide solution is above the temperature of standardization. the percent sulfuric acid as follows:

Percent sulfuric acid = $\frac{4.904 \text{ AN}}{W}$

where: A = corrected volume of sodium hydroxide solution, in ml.,

> N = normality of sodium hydroxide solution, and

W = weight of sample in grams.

4.6.4 Specific gravity (see 6.3). Determine the specific gravity using a suitable precision hydrometer standardized at 60°F., referred to water at 60°F. Make temperature corrections, when necessary, for deviations from the standard temperature as shown in table II. When the temperature of the acid is above the standard temperature, the correction is added to the observed specific gravity value; when the temperature of the acid is below the standard temperature, the correction is subtracted from the observed specific gravity value.

TABLE II.—Correction for temperature deviation

• · · · · · · · · · · · · · · · · · · ·	Correction per degree of
Acid	temperature deviation
Class 1	0.00054
Class 2	0.00041
Class 3	0.00039
Class 4	0.00036

4.6.5 Impurities.

4.6.5.1 Organic matter. Heat 50 ml. of the acid in a clean beaker until the acid begins to fume strongly. To pass test, there shall be no perceptible charring; a slightyellow color that appears when the acid is hot but disappears on cooling should be disregarded.

4.6.5.2 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 acid 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 aqua 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 5or 6 drops. (If the solution accidentally evaporates completely, repeat the digestion with aqua regia and reevaporate to 5 or 6 drops.) Absorb the solution (5 or 6 drops) in a piece of thin asbestos paper (preferably not over 0.75 mm. thick) 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 steam 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.)

4.6.5.3 Fixed residue. As applicable, measure 10 ml. of class 1, 24 ml. of class 2, 36 ml. of class 3, or 52 ml. of class 4 acid into a platinum dish, evaporate to dryness, and ignite at a dull red heat for 5 minutes. Cool in a dessiccator and weigh

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the residue (save the residue for iron and copper determinations); calculate the percent fixed residue as follows:

Percent fixed residue = $\frac{W}{V \times S}$ where: W = weight of residue in grams,

- V = volume of acid specimen in milliliters, and
- S = specific gravity of acid specimen (see 4.6.4).

4.6.5.4 Sulfurous acid. As applicable, dilute 20 ml. of class 1, 49 ml. of class 2, or 72 ml. of class 3 acid to 80 ml. with water; in the case of the class 4 acid, 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 0.1N potassium permanganate solution to a faint but permanent pink color. No more than 0.46 ml. of 0.1N potassium permanganate solution shall be required. Save the titrated solution for 4.6.5.5.

4.6.5.5 Preliminary test for antimony, copper. and selenium. Dilute the titrated solution from 4.6.5.4 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 centimeter (cm.) quantitative filter paper of close texture (save the filtrate for 4.6.5.12). 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 hydrogen sulfide treatment, the preliminary test for antimony may be meaningless. The precipitation of sulfur will not obscure copper or selenium, if present.)

4.6.5.6 Iron. (If the percent fixed residue, 4.6.5.3, 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 (4.6.5.3), warm on a steam bath to dissolve 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 milligrams (mg.) of ammonium persulfate, and 3 ml. of a 30percent 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.

4.6.5.7 Copper. (If the percent fixed residue, 4.6.5.3, 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, 4.6.5.5, does not indicate the presence of copper; the determination of copper is to be omitted.) Neutralize the remaining 9 ml. of solution A (4.6.5.6) 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.

4.6.5.8 Zinc. (If the percent fixed residue, 4.6.5.3, does not exceed 0.0060 for class 1, 0.0032 for class 2, 0.0024 for class 3, or 0.0019 for class 4; the 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 acid to approxiDownloaded from http://www.everyspec.com

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mately 110 ml. and neutralize with ammonium hydroxide. Dilute the solution to 200 ml., add 10 ml. of 6N 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. Pipet 1 ml. of 1N 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 in a dessiccator, and weigh the collected precipitate. Calculate the percent zinc as follows:

Percent zinc =
$$\frac{W}{V \times S}$$

- where: W = weight of collected precipitate in grams,
 - V = volume of acid specimen in milliliters, and
 - S = specific gravity of acid specimen (see 4.6.4).

4.6.5.9 Arsenic. As applicable, dilute 20 ml. of class 1, 49 ml. of class 2, 58 ml. of class 3, or 78 ml. of class 4 acid to 100 ml. in a volumetric flask. As applicable, dilute a 20-ml. aliquot of the class 1 or class 2 acid dilution or a 25-ml. aliquot of the class 3 or class 4 acid dilution to 50 ml. and determine arsenic by the modified Gutzeit procedure. The stain produced shall not exceed that produced by 0.0075 mg. of arsenic. (Note: Also compare stain with that produced by 0.00075 mg. of arsenic in reference to 4.6.5.10.)

4.6.5.10 Antimony. If the arsenic content has been found to be not greater than one tenth of the permitted limit (see note of 4.6.5.9), antimony is determined without first removing arsenic; proceed as in 4.6.5.10.1. If the arsenic content has been found greater than one-tenth the permitted limit, proceed as in 4.6.5.10.2.

4.6.5.10.1 (Arsenic, 0.00001 percent or less.) As applicable, evaporate 11 ml. of class 1, 27 ml. of class 2, 40 ml. of class 3, or 57 ml. of class 4 acid 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.

4.6.5.10.2 (Arsenic greater than 0.00001 percent.) As applicable, evaporate 55 ml. of class 1, 110 ml. of class 2, 162 ml. of class 3, or 225 ml. of class 4 acid to 4 or 5 ml. in a 250-ml. beaker. Cool 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 to 45 ml., keeping the temperature of the liquid below 110°C. (use boiling tubes or chips to minimize bumping-take care not to let the volume get too small or the temperature get too high; otherwise some antimony may be lost). Transfer quantitatively, with a minimum amount of water, the reduced volume 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 directed in The stain developed shall not 4.6.5.10.1. exceed that produced by a solution containing 0.02 mg. antimony, 3 ml. of reagent sulfuric acid, and 4 ml. of hydrochloric acid.

4.6.5.11 Selenium. (If the preliminary test for selenium, 4.6.5.5, was negative, the determination of selenium is to be omitted.)

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Classes 2, 3, and 4 acids require 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 acid 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 with the remaining portion of the 10 ml. of hydrochloric acid.) Observe the zone of contact between the two acids for 3 minutes from the time the sample is completely covered by the first addition of hydrochloric acid. The formation of a red to reddish-brown ring denotes the presence of a 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 reddishbrown 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 confused with the yellow ring.)

4.6.5.12 Nickel. Gently boil 50 ml. of the filtrate saved from 4.6.5.5 to remove the excess hydrogen sulfide. Cool, add 2 ml. of a 20-percent tartaric acid solution and 2 ml. of water saturated with bromine. Slowly add ammonium hydroxide until the bromine color is discharged. Cool the solution to room temperature. Add 2 ml. of ammonium hydroxide, 2 ml. of a 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 filtrate sample after boiling.

4.6.5.13 *Manganese*. As applicable, dilute 20 ml. of class 1, 49 ml. of class 2, or 72 ml. of class 3 acid to 95 ml. with water and

add 5 ml. of nitric acid; in the case of class 4 acid, 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; the color shall not be deeper than that of a standard containing 0.7 ml. of 0.001N potassium permanganate diluted to 95 ml. with water and treated in the same manner.

4.6.5.14 Nitrate. 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 pipetting 20 ml. of the stock solution into a 1-liter volumetric flask and make up to volume.

1 ml. of standard = 0.0125 mg. KNO₃ = 0.00766 mg. NO₃

4.6.5.14.1 Class 1. Pipet 10 ml. of water into one of a matched pair of Nessler tubes and add, by pipet, 5 ml. of class 1 acid and 15 ml. of nitrate-free reagant sulfuric acid. Into the second Nessler tube, pipet 4 ml. of water and 6 ml. of the standard nitrate solution (4.6.5.14); 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 acid 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.)

4.6.5.14.2 Class 2. Pipet 10 ml. of class 2 acid into one of a matched pair of Nessler tubes and add, by pipet, 10 ml. of nitrate-free sulfuric acid. Into the second Nessler tube, pipet 5 ml. of the standard nitrate solution (4.6.5.14) 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 acid shall not exceed that of the solution in the Nessler tube containing the standard nitrate solution. (Note: See note of 4.6.5.14.1.)

4.6.5.14.3 Class 3. Pipet 15 ml. of class 3 acid into one of a matched pair of Nessler tubes and add, by pipet, 5 ml. of nitrate-free sulfuric acid. Into the second Nessler tube, pipet 5 ml. of the standard nitrate solution (4.6.5.14) 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 acid shall not exceed that of the solution in the Nessler tube containing the standard nitrate solution. (Note: See note of 4.6.5.14.1.)

4.6.5.14.4 Class 4. Pipet 20 ml. of class 4 acid into one of a matched pair of Nessler tubes. Into the second Nessler tube. pipet 5 ml. of water and 5 ml. of the standard nitrate solution (4.6.5.14); 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 acid shall not exceed that of the solution in the Nessler tube containing the standard nitrate solu-(Note: See note of 4.6.5.14.1.) tion.

4.6.5.15 Ammonium. As applicable, dilute 20 ml. of class 1, 49 ml. of class 2, 58 ml. of class 3, or 78 ml. of class 4 acid to 100 ml. in a volumetric flask. As applicable, in a Nessler tube, dilute 20 ml. of the class 1 or class 2 acid dilution or dilute 25 ml. of the class 3 or class 4 acid 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 ion (1 ml. of a 1-liter solution of 0.22 g. of ammonium chloride in water) treated in the same manner.

4.6.5.16 Chloride. As applicable dilute 20 ml. of class 1, 49 ml. of class 2, 36 ml. of class 3, or 49 ml. of class 4 to 100 ml. in a volumetric flask. As applicable, in a Nessler tube, dilute 5 ml. of class 1 or class 2 acid dilution or dilute 10 ml. of class 3 or class 4 acid 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 a 1-liter solution of 0.030 g. of sodium chloride in water) treated in the same manner.

5. PREPARATION FOR DELIVERY

For civil agency procurement, the definitions and applications of the levels of packaging and packing shall be in accordance with Fed. Std. No. 102.

5.1 Packaging. Packaging shall be level A or C as specified, (see 6.2).

5.1.1 Level A. Sulfuric acid, electrolyte shall be packaged in 1-gallon quantities in accordance with MIL-S-207.

5.1.2 Level C. Sulfuric acid, electrolyte shall be packaged to afford adequate protection from the supply source to the first receiving activity for immediate use.

5.2 Packing. Packing shall be level A, B, or C, as specified (see 6.2).

5.2.1 Level A.

5.2.1.1 One-gallon quantities. One-gallon quantities of sulfuric acid, electroylte shall be packed in accordance with MIL-S-207.

5.2.1.2 F i v e-to-fiftcen-gallon quantities. Sulfuric acid, electrolyte shall be packed, as specified (see 6.2), in 5-, $6\frac{1}{2}$ -, or 13-

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gallon glass carboys conforming to Specification 1A of Interstate Commerce Commission Regulations for Transportation of Explosive and Other Dangerous Articles, or 5- or 15-gallon polyethylene lined drums conforming to MIL-D-40030. Use of polyethylene-lined drums shall be limited to class 3 and class 4 sulfuric acid, electrolyte.

5.2.2 Level B. One-gallon quantities of class 3 or class 4 sulfuric acid, electrolyte, packaged in polyethylene bottles in accordance with types III or IV of MIL-S-207 shall be packed in accordance with the level B requirements for type III of MIL-S-207.

5.2.3 Level C. Sulfuric acid, electrolyte shall be packed to insure carrier acceptance and safe delivery at first domestic destination. Containers shall be in accordance with Interstate Commerce Commission Regulations or other regulations applicable to the mode of transportation.

5.3 Marking.

5.3.1 *Civil agencies.* In addition to regulatory and precautionary marking and any special marking required by the contract or order, shipments shall be marked in accordance with Fed. Std. No. 123.

5.3.2 Military agencies. In addition to regulatory and precautionary marking and any special marking required by the contract or order, shipments shall be marked in accordance with MIL-STD-129.

5.3.3 Regulatory marking. Each shipping container shall be labeled as required by Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles.

5.3.4 Precautionary marking. Each container shall be marked to meet the requirements of MIL-STD-129 with the following: DANGER: CAUSES SEVERE BURNS

Do not get IN EYES, ON SKIN or clothing.

In case of contact, immediately flush skin or eyes with plenty of water for at least 15 minutes; for eyes, GET MEDICAL ATTENTION.

Do not add water to contents while in a container because of violent reaction.

6. NOTES

6.1 Intended use. Sulfuric acid, electrolyte, covered by this specification, is intended for use in electrolytic solutions for lead storage batteries. Class 1, concentrated sulfuric acid, should be diluted to the applicable density specified for class 2, 3, or 4, respectively, before use; it should never be used undiluted in lead storage batteries.

6.2 Ordering data. Purchasers should exercise any desired options offered herein and procurement documents should specify the following:

- (a) Number, title, and date of this specification.
- (b) Class of acid required.
- (c) Level of packaging or packing required.
- (d) Quantity per package or container required.

6.3 Specific gravity equivalency. The specific gravity of a solution will vary with temperature. Table III covers specific gravity equivalency at several temperatures for those users and suppliers who prefer temperature basis other than 60°F. For test purposes at other than 60°F., an appropriately standardized hydrometer will be required.

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TABLE III.—Specific gravity equivalency

	Specific gravity						
Class	@ 60°F/60°F		@ 77°F/77°F		@ 80°F/80°F		
	Min.	Max.	Min.	Max.	Min.	Max.	
1	1.8354		·	<u> </u>	1.828		
2	1.3945	1.4042	_		1.3895	1.8991	
3	1.2767	1.2853	_	—	1.2720	1.2806	
4	1.2085	1.2185	1.2050	1.2150	1.2044	1.2144	

6.4 Batch. A batch is defined as that quantity of material which 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 Significant places. For the purpose of determining conformance with this specification, an observed or a calculated value shall 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 the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E29).

6.6 Military use. Items procured under this specification for military use are to be limited to the variety shown on the applicable military standards or military supply standard. Personnel of the military departments are requested to refer to these documents for guidance.

6.7 Transportation description. Trans-

portation descriptions and minimum weights applicable to this commodity are:

Classes 1 and 2

Rail:

Sulfuric acid.

Carload minimum weights 30,000 and 36,000 pounds.

Motor:

Sulfuric acid.

Truckload minimum weights 30,000 and 36,000 pounds, subject to Rule 115, National Motor Freight Classification.

Classes 3 and 4

Rail:

Acids, electrolyte, containing not to exceed 47 percent sulfuric acid.

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Carload minimum weight 30,000 pounds.

Motor:

Acids, electrolyte, containing not to exceed 47 percent sulfuric acid.

Truckload minimum weight 30,000 pounds, subject to Rule 115, National Motor Freight Classification.

MILITARY CUSTODIANS:

Army-MU

Navy-SH

Air Force----

Review interest:

Army-MO

Navy-YD

User interest:

Navy-WP

Preparing activity:

Army—MU(EA)

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