

O-S-801C
October 20, 1978
SUPERSEDING
Fed. Spec. O-S-801B
April 14, 1965 and
MIL-S-207B
November 3, 1965

FEDERAL SPECIFICATION

SULFURIC ACID, ELECTROLYTE; FOR STORAGE BATTERIES

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

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers four classes of sulfuric acid for use in storage batteries.

1.2 Classification. Sulfuric acid, electrolyte shall be of the following classes as specified (see 6.2):

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

2. APPLICABLE DOCUMENTS

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

Federal Specifications:

- L-P-390 - Plastic, Molding and Extrusion Material, Polyethylene and Copolymers (Low, Medium, and High Density).
- HH-I-585 - Insulation, Thermal.
- NN-P-71 - Pallets, Material Handling, Wood, Stringer Construction, 2-Way and 4-Way (Partial).

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- QQ-S-781 - Strapping, Steel, and Seals.
- PPP-B-585 - Boxes, Wood, Wirebound.
- PPP-B-601 - Boxes, Wood, Cleated-Plywood.
- PPP-B-621 - Boxes, Wood, Nailed and Lock-Corner.
- PPP-B-636 - Boxes, Shipping, Fiberboard.
- PPP-B-640 - Boxes, Fiberboard, Corrugated, Triple-Wall.
- PPP-B-1163 - Box, Corrugated Fiberboard, High Compression Strength, Weather-Resistant, Wax-Resin Impregnated.
- PPP-C-186 - Containers, Packaging and Packing for Drugs, Chemicals, and Pharmaceuticals.
- PPP-C-569 - Containers, Plastic, Molded (For Liquids, Pastes, and Powders): Overpacked.
- PPP-C-1337 - Containers, Composite; (Steel Drum With Polyethylene Insert).
- PPP-F-320 - Fiberboard; Corrugated and Solid, Sheet Stock (Container Grade), and Cut Shapes.
- PPP-T-66 - Tape: Pressure-Sensitive Adhesive, Vinyl Plastic Film.
- PPP-T-97 - Tape, Pressure-Sensitive Adhesive, Filament Reinforced.

Federal Standards:

- Fed. Test Method Std. No. 101 - Preservation, Packaging, and Packing Materials: Test Procedures.
- Fed. Std. No. 123 - Marking for Shipment (Civil 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 and 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, US Government Printing Office, Washington, DC 20402.

(Single copies of this specification and other Federal specifications required by activities outside the Federal Government for bidding purposes are available without charge from Business Service Centers at the General Services Administration Regional Offices in Boston, New York, Philadelphia, Washington, DC, Atlanta, Chicago, Kansas City, MO, Fort Worth, Houston, Denver, San Francisco, Los Angeles, and Seattle, WA.

(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

Military Specifications:

- MIL-C-5501/7 - Caps and Plugs, Protective, Dust and Moisture Seal
(Cap-Plug, General Purpose).
- MIL-D-43703 - Drums, Shipping and Storage, Molded Polyethylene.

Military Standards:

- MIL-STD-105 - Sampling Procedures and Tables for Inspection by
Attributes.
- MIL-STD-129 - Marking for Shipment and Storage.
- MIL-STD-147 - Palletized Unit Loads for 40" by 48" Pallets.
- MIL-STD-1188 - Commercial Packaging of Supplies and Equipment.

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

Laws and Regulations:

- 49 CFR 171 to 179 - Department of Transportation Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles.

(The Department of Transportation regulations are a part of the Code of Federal Regulations available from the Superintendent of Documents, US Government Printing Office, Washington, DC 20402. Orders for the above publications should cite "49 CFR 171 to 179.")

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.)

Society of Plastics Industry, Inc.:

- SP-400 Finish For Plastic Bottles

(Application for copies should be addressed to Seajay, Inc., Apple Street, New Shrewsbury, NJ 07724.)

3. REQUIREMENTS

3.1 Material. Sulfuric acid shall be nonfuming and free from sediment when tested as specified in 4.2.4.1.

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3.2 Color. Sulfuric acid shall have a color no darker than that of a standard color solution when tested as specified in 4.2.4.2.

3.3 Acid content and specific gravity. Sulfuric acid shall conform to the applicable characteristics of table I when tested for acid content as specified in 4.2.4.3 and specific gravity at 60°/60° F as specified in 4.2.4.4.

TABLE I. Acid content and specific gravity

| Class | : Acid content, percent by weight : | | : Specific gravity at 60°/60° F | |
|---------|-------------------------------------|-----------|---------------------------------|-----------|
| | : Minimum | : Maximum | : Minimum | : Maximum |
| Class 1 | : 93.2 | : ----- | : 1.8354 | : ----- |
| Class 2 | : 49.5 | : 50.5 | : 1.3945 | : 1.4042 |
| Class 3 | : 36.5 | : 37.5 | : 1.2767 | : 1.2853 |
| Class 4 | : 28.3 | : 29.5 | : 1.2085 | : 1.2185 |

3.4 Impurities. Sulfuric acid shall conform to the maximum impurities characteristics of table II when tested as specified therein.

TABLE II. Maximum impurities

| Impurity | : Maximum percent by weight | | | | : Test paragraph |
|----------------|-----------------------------|------------|------------|------------|------------------|
| | : Class 1 | : Class 2 | : Class 3 | : Class 4 | |
| Organic matter | : * | : * | : * | : * | : 4.2.4.5 |
| Platinum | : * | : * | : * | : * | : 4.2.4.6 |
| Fixed residue | : 0.03 | : 0.016 | : 0.012 | : 0.009 | : 4.2.4.7 |
| Sulfurous acid | : 0.004 | : 0.0022 | : 0.0016 | : 0.0013 | : 4.2.4.8 |
| Iron | : 0.005 | : 0.0027 | : 0.0020 | : 0.0016 | : 4.2.4.10 |
| Copper | : 0.005 | : 0.0027 | : 0.0020 | : 0.0016 | : 4.2.4.11** |
| Zinc | : 0.004 | : 0.0022 | : 0.0016 | : 0.0013 | : 4.2.4.12 |
| Arsenic | : 0.0001 | : 0.00005 | : 0.00004 | : 0.00003 | : 4.2.4.13 |
| Antimony | : 0.0001 | : 0.00005 | : 0.00004 | : 0.00003 | : 4.2.4.14** |
| Selenium | : 0.002 | : 0.0011 | : 0.0008 | : 0.0006 | : 4.2.4.15** |
| Nickel | : 0.0001 | : 0.00005 | : 0.00004 | : 0.00003 | : 4.2.4.16 |
| Manganese | : 0.00002 | : 0.000011 | : 0.000008 | : 0.000006 | : 4.2.4.17 |
| Nitrate | : 0.0005 | : 0.00027 | : 0.00020 | : 0.00016 | : 4.2.4.18 |
| Ammonium | : 0.001 | : 0.0005 | : 0.0004 | : 0.0003 | : 4.2.4.19 |
| Chloride | : 0.001 | : 0.0005 | : 0.0004 | : 0.0003 | : 4.2.4.20 |

*To pass test.

**See 4.2.4.9 for preliminary test.

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4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or 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 the specification where such inspections are deemed necessary to assure that supplies and services conform to the specified requirements.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the sulfuric acid, electrolyte offered for acceptance at one time which has been produced by one manufacturer, at one plant, during one shift, from 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 6.3).

4.2.2 Sampling.

4.2.2.1 For sulfuric acid tests. 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.

TABLE III. Sampling for sulfuric acid tests

| 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 |
| | : | |

4.2.2.2 For examination of preparation for delivery and container tests. Sampling shall be conducted in accordance with table IV.

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TABLE IV. Sampling for examination of preparation for delivery and container tests

| Examination or test | : MIL-STD-105, : AQL, |
|---|------------------------|
| | : inspection : percent |
| | : level : defective |
| Examination of preparation for delivery | : S-3 : 2.5 |
| Container leakage test | : S-4 : 1.5 |
| Container contamination test | : S-3 : 1.5 |
| Container shock resistance test | : S-1 : 4.0 |

4.2.3 Inspection procedure.

4.2.3.1 For sulfuric acid tests. Each specimen taken in 4.2.2.1 shall be tested as specified in 4.2.4. Failure of any test by any specimen shall be cause for rejection of the lot represented.

4.2.3.2 For examination of preparation for delivery. The sample unit shall be one filled unit, intermediate, or shipping container, as applicable, ready for shipment. Sample unit, intermediate, and shipping containers shall be examined for the following defects using inspection level S-3 and an AQL of 2.5 percent defective:

- (a) Contents per container not as specified
- (b) Container not as specified
- (c) Container closure not as specified or not tight
- (d) Container damaged or leaking
- (e) Plug missing, not as specified, or improperly seated in DOT 2U container closure neck
- (f) Spout not extendable from DOT 2U container
- (g) Spout of DOT 2U container not easily accessible
- (h) Reinforcing straps or tape missing, loose, not as specified, or causing damage to the container (where required)
- (i) Cushioning material not as specified or missing (where required)
- (j) Fiberboard packing aids missing, not as specified, or improperly placed (where required)
- (k) Marking incorrect, missing, illegible, or not durable
- (l) Palletization not as specified

4.2.3.3 For container tests.

(a) Leakage. The sample unit shall be one filled and closed unit container. Sample unit containers shall be selected in accordance with table IV and tested for leakage as specified in 4.2.5.1.

(b) Contamination. The sample unit shall be one filled and closed 1-gallon polyethylene bottle in its fiberboard box. Sample containers shall be selected in accordance with table IV and tested for contamination as specified in 4.2.5.2.

(c) Shock resistance. The sample unit shall be one filled and closed intermediate fiberboard box containing a filled and closed 1-gallon polyethylene bottle. Sample containers shall be selected in accordance with table IV and tested for shock resistance as specified in 4.2.5.3.

4.2.4 Sulfuric acid tests. Water in accordance with 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:

4.2.4.1 Material. Visually examine the specimen for evidence of fuming and for the presence of sediment.

4.2.4.2 Color. Compare the color of 50 milliliters (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 milligrams (mg) of copper, 1 ml of 0.1N hydrochloric acid, and 5 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 approximate dimensions of 30 centimeters (cm) in length by 2 cm in diameter.

4.2.4.3 Acid content. 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 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 in 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

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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 or by subtracting 0.00035 ml per milliliter of titer for each degree C that the sodium hydroxide solution is above the temperature of standardization. Calculate the percent by weight sulfuric acid as follows:

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

where: A = Corrected milliliters of sodium hydroxide solution used in the titration,
 B = Normality of the sodium hydroxide solution, and
 W = Weight of specimen in grams.

4.2.4.4 Specific gravity. Determine the specific gravity of the specimen 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 V. When the temperature of the acid is above the standard temperature, add the correction to the observed specific gravity value; when the temperature of the acid is below the standard temperature, subtract the correction from the observed specific gravity value.

TABLE V. Correction for temperature deviation

| Class of sulfuric acid | : | Correction per degree F of temperature deviation |
|------------------------|---|--|
| | : | |
| Class 1 | : | 0.00054 |
| Class 2 | : | 0.00041 |
| Class 3 | : | 0.00039 |
| Class 4 | : | 0.00036 |
| | : | |

4.2.4.5 Organic matter. 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.

4.2.4.6 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 aqua regia (4 volumes of hydrochloric acid and 1 volume of nitric acid). Cover the

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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 aqua 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 millimeter (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.

4.2.4.7 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 specimen into a platinum dish, evaporate to dryness, and ignite at a dull red heat for 5 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:

$$\text{Percent fixed residue} = \frac{A}{BC}$$

where: A = Weight of residue in grams,
 B = Milliliters of specimen, and
 C = Specific gravity of the specimen determined in 4.2.4.4.

4.2.4.8 Sulfurous acid. 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 the 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 0.1N 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 use in 4.2.4.9.

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4.2.4.9 Preliminary test for antimony, copper, and selenium. Dilute the titrated solution from 4.2.4.8 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 of close texture (save the filtrate for 4.2.4.16). 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.)

4.2.4.10 Iron. (If the percent fixed residue determined in 4.2.4.7 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 from 4.2.4.7, 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.

4.2.4.11 Copper. (If the percent fixed residue determined in 4.2.4.7 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 4.2.4.9 does not indicate the presence of copper; the determination of copper is to be omitted). Neutralize the remaining 9 ml of solution A from 4.2.4.10 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.

4.2.4.12 Zinc. (If the percent fixed residue determined in 4.2.4.7 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,

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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 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 to room temperature in a desiccator, and weigh the collected precipitate. Calculate the percent by weight zinc as follows:

$$\text{Percent zinc} = \frac{A}{BC}$$

where: A = Weight of collected precipitate in grams,

B = Milliliters of specimen, and

C = Specific gravity of specimen determined in 4.2.4.4.

4.2.4.13 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 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 the 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 to 4.2.4.14.)

4.2.4.14 Antimony. If the arsenic content has been found to be not greater than one-tenth of the permitted limit (see note of 4.2.4.13), antimony is determined without first removing arsenic; proceed as specified in (a) below. If the arsenic content has been found greater than one-tenth the permitted limit, proceed as specified in (b) below.

(a) Procedure when arsenic content is 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 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° ± 3° 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.

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(b) Procedure when arsenic content is 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 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 to 45 ml keeping the temperature of the liquid below 110° C. (Use boiling tubes or chips to minimize bumping and take care not to let the volume get too small or the temperature get 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 (a). The stain developed shall not exceed that produced by a solution containing 0.02 mg antimony, 3 ml of reagent sulfuric acid, and 4 ml of hydrochloric acid.

4.2.4.15 Selenium. (If the preliminary test for selenium in 4.2.4.9 was negative, the determination of selenium is to be omitted.) Classes 2, 3, and 4 specimens 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 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 with 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 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 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.)

4.2.4.16 Nickel. Gently boil 50 ml of the filtrate retained from 4.2.4.9 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 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 specimen solution filtrate after boiling.

4.2.4.17 Manganese. 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 0.001N potassium permanganate solution diluted to 95 ml with water and treated in the same manner.

4.2.4.18 Nitrate.

(a) Standard nitrate solution. 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.)

(b) 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 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 (a). 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.)

(c) Class 2. 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 sulfuric acid. Into the second Nessler tube, pipet 5 ml of the standard nitrate solution prepared in (a) and add, by pipet, 15 ml of nitrate-free reagent sulfuric acid. To each Nessler tube, add 0.5 ml of a saturated

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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 (b).

(d) Class 3. 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 sulfuric acid. Into the second Nessler tube, pipet 5 ml of the standard nitrate solution prepared in (a) 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 (b).

(e) Class 4. Pipet 20 ml of class 4 specimen 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 prepared in (a) 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 (b).

4.2.4.19 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 specimen to 100 ml in a volumetric flask. As applicable, in a Nessler tube, dilute 20 ml of the class 1 or class 2 specimen dilution or dilute 25 ml of the 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 ion (1 ml of a 1-liter solution of 0.22 g of ammonium chloride in water) treated in the same manner.

4.2.4.20 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 specimen to 100 ml in a volumetric flask. As applicable, in a Nessler tube, dilute 5 ml of the 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 a 1-liter solution of 0.030 g of sodium chloride in water) treated in the same manner.

4.2.5 Container tests. Tests shall be conducted as follows:

4.2.5.1 Leakage. Place the container in an inverted position at a temperature of $75^{\circ} \pm 5^{\circ}$ F for 4 hours. Observe the container for evidence of leakage during and at the end of this test.

4.2.5.2 Contamination. Use clean, contamination-free gloves during the testing. Wipe the entire exterior surface of the polyethylene container and the exposed surfaces of the box flaps with neutral or blue litmus indicator paper. Special attention should be paid to the area around the seams and the plug closure in the spout. Red coloration of the litmus paper shall be considered evidence of sulfuric acid contamination.

4.2.5.3 Shock resistance. Condition the container for 72 hours at $-40^{\circ} \pm 2^{\circ}$ F prior to testing. Subject the container to a free fall drop test in accordance with procedures B and E, method 5007 of Fed. Test Method Std. No. 101 until the drop cycle is completed or failure occurs. After the test has been completed, invert the container and allow it to remain in this position for 24 hours before opening to inspect for leakage.

5. PREPARATION FOR DELIVERY

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

5.1.1 Level A.

5.1.1.1 Five-pint quantity, class 1 sulfuric acid. A quantity of 5 pints [± 1 or -0 fluid ounces (oz)] of class 1 sulfuric acid shall be packaged in a nominal 5-pint capacity glass bottle in accordance with Department of Transportation (DOT) regulations. The bottle shall conform to group A, class 1, type d, grade 2, closure A, seal A of PPP-C-186. The closure A of PPP-C-186 shall be made of styrene-acrylonitrile or a material of equal or better durability in this service. The closure shall be secured to the neck of the bottle by tightening to a torque of 18 to 25 pound-inches. Seal A of PPP-C-186 shall secure the screw-cap to the neck of the bottle by the circumferential application of a minimum of 1-1/3 turns of tape conforming to type optional of PPP-T-66. The tape shall extend from the top of the cap to well down around the neck of the bottle. Alternatively, a self-shrinking outerseal meeting the minimum performance requirements of this tape shall be acceptable. The closed bottle shall not leak when tested as specified in 4.2.5.1. The closed

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bottle shall be placed upright in an expanded polystyrene case in accordance with DOT Specification 33A and the case shall be closed for shipment by using waterproof tape as specified therein. The polystyrene case shall then be overpacked in a close-fitting fiberboard box conforming to grade W5c of PPP-B-636.

5.1.1.2 One-gallon (gal) quantity.

(a) Class 1 sulfuric acid. A quantity of 1 US gal (+1 or -0 oz) of class 1 sulfuric acid shall be unit packaged as specified in 5.1.1.1 except that the unit container shall be a nominal 1-gal capacity glass bottle or jug. The closed bottle or jug shall not leak when tested as specified in 4.2.5.1. The closed bottle or jug shall be centered upright in a fiberboard box conforming to type CF, class domestic, variety DW, grade 200 of PPP-B-636 and having minimum inside dimensions of 10 by 10 by 17 inches. Vermiculite conforming to type IV, class 1 of HH-I-585 shall be added to protect all surfaces of the bottle or jug and to completely fill the box. The box shall be closed by taping all seams as specified in PPP-B-636.

(b) Classes 2, 3, and 4 sulfuric acid. A quantity of 1 US gal (+1 or -0 oz) of class 2, 3, or 4 sulfuric acid as specified (see 6.2) shall be packaged in a nominal 1-gal capacity rectangular polyethylene bottle conforming to DOT Specification 2U in a fiberboard box conforming to type I of PPP-C-569 except that the bursting strength shall be no less than 200 pounds per square inch. Each bottle shall be furnished with a nonintegral spout which has been injection molded from virgin natural polyethylene conforming to type I, class H, grade optional or type I, class M, grade 2 of L-P-390. The spout shall be designed and sized to conform to Glass Container Manufacturer's Institute Standards as modified by the Society of the Plastics Industry, Incorporated for a nominal 38-millimeter (mm) diameter screw cap with a minimum of one and one-half turns of a buttress thread. The inner surface and top rim of the spout shall be properly sized and sufficiently smooth and concentric to form a leakproof mating surface with the plug which is inserted after filling. A snug-fitting plug conforming to MIL-C-5501/7 shall be inserted into the opening of the spout of the filled bottle until the underside of the shoulder of the plug is in full contact with the top rim of the spout. Before applying the screw-cap, the exterior of the filled bottle shall show no evidence of contamination by sulfuric acid when tested as specified in 4.2.5.2. The plug shall then be secured tightly in place in the spout by a nominal 38-mm diameter phenolic buttress screw-thread cap designed to match the spout. The fiberboard box shall be perforated to provide for easy removal of a section to permit ready access to and extension of the pouring spout. The box shall be closed as specified in the appendix to PPP-C-569. The filled and closed box shall show no evidence of leakage when tested for shock resistance as specified in 4.2.5.3.

5.1.2 Level B.

5.1.2.1 One-gal quantity, classes 2, 3, and 4 sulfuric acid. A quantity of 1 US gal (+1 or -0 oz) of class 2, 3, or 4 sulfuric acid as specified (see 6.2) shall be packaged as specified in 5.1.1.2(b) except that the fiberboard box shall conform to the DOT Specification 12B special box for DOT Specification 2U containers not over 5-gal in capacity, and the box shall be formed from board having a classified completed strength of no less than 200 pounds. The fiberboard box shall be perforated to provide for easy removal of a section to permit ready access to and extension of the pouring spout. The polyethylene container shall be tested for contamination and the fiberboard box shall be tested for shock resistance as specified in 5.1.1.2(b).

5.1.3 Level C.

5.1.3.1 Civil agencies. A quantity of sulfuric acid as specified (see 6.2) shall be packaged to assure carrier acceptance and safe delivery to the first domestic destination. Containers shall be in accordance with DOT regulations and applicable carrier regulations.

5.1.3.2 Military activities. A quantity of sulfuric acid as specified (see 6.2) shall be packaged in accordance with MIL-STD-1188.

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

5.2.1 Level A.

5.2.1.1 Five-pint quantity, class 1 sulfuric acid. Four single bottle cases of class 1 sulfuric acid packaged as specified in 5.1.1.1 shall be packed upright in accordance with DOT regulations in a close-fitting wood box conforming to class 2, grade A, style 4 of PPP-621; type overseas, style A, B, or I, grade A of PPP-B-601; or class 3, style optional of PPP-B-585 with wood preserved as specified for grade A of PPP-B-621. The box shall be for a type 2 load. Motion of contents shall be prevented by the insertion, where needed, of fiberboard pads formed from material conforming to grade V3c of PPP-F-320. The box shall be closed and reinforced as specified in the appendix to the applicable box specification.

5.2.1.2 One-gal quantity.

(a) Class 1 sulfuric acid. Four 1-gal containers of class 1 sulfuric acid packaged as specified in 5.1.1.2(a) shall be packed upright in a wood box as specified in 5.2.1.1.

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(b) Classes 2, 3, and 4 sulfuric acid. Four packages of class 2, 3, or 4 sulfuric prepared as specified in 5.1.1.2(b) shall be packed upright in a close-fitting impregnated fiberboard box conforming to style RSC, class I, type SWCFI, grade 275 of PPP-B-1163. When specified (see 6.2), the four packages of class 2, 3, or 4 sulfuric acid shall be packed upright in a close-fitting cleated fiberboard box conforming to class 2, style A of PPP-B-640. Motion of contents shall be prevented by the insertion, where needed, of the same fiberboard as that used for the box. The box shall be closed by water-resistant adhesive plus staples and the box shall be reinforced by strapping as specified in the appendix to the box specification. If staples are used, fiberboard pads shall be inserted, where needed, to prevent the staples from puncturing the inner containers. The containers shall show no evidence of leakage of contents when tested as specified in 4.2.5.1.

5.2.1.3 Five-gal quantity, class 2 sulfuric acid. A quantity of 5 US gal (+6 or -0 oz) of class 2 sulfuric acid shall be packed in accordance with DOT regulations in a nominal 5-gal capacity container conforming to type II, class 1 of PPP-C-1337 or DOT Specification 34 as specified (see 6.2). The container shall show no evidence of leakage of contents when tested as specified in 4.2.5.1.

5.2.1.4 Six and one-half-gal quantity, class 1 sulfuric acid. A quantity of 6-1/2 US gal (+8 or -0 oz) of class 1 sulfuric acid shall be packed in accordance with DOT regulations in a nominal 6-1/2-gal capacity glass carboy conforming to DOT Specification 1A, 1C, or 1K or in a glass carboy totally enclosed by an outside container formed from foamed polystyrene and permitted by DOT as an exception to existing specifications as specified (see 6.2). The outside container shall completely enclose the closure neck and body of the glass carboy. The closure of the outside container shall be reinforced by no less than two steel straps of 1/2-inch minimum width conforming to class 1, type optional of QQ-S-781 applied vertically and at right angles to each other. The outside polystyrene container closure shall be secured by tape conforming to type IV of PPP-T-97 rather than by the steel straps specified above. One continuous strip of the tape shall encircle and be vertically centered over the surface of the polystyrene container. Another continuous strip of the tape, no less than 3 inches wide, shall encircle the case and be centered over the seam between the top and bottom sections. The strips of tape shall overlap no less than 3 inches. The containers shall show no evidence of leakage of contents when tested as specified in 4.2.5.1.

5.2.1.5 Fifteen-gal quantity, class 3 sulfuric acid. A quantity of 15 US gal (+1 or -0 pint) of class 3 sulfuric acid shall be packed in a nominal 15-gal container conforming to type II, class 2A of PPP-C-1337 or size II of MIL-D-43703 as specified (see 6.2) except that aside from the requirement for rolling rings, the shape and dimensions of the drum shall not be restricted to that specified, but shall be restricted to any compact configuration meeting the specified performance requirements and DOT regulations. The container shall show no evidence of leakage of contents when tested as specified in 4.2.5.1.

5.2.2 Level B.

5.2.2.1 Five-pint quantity, class 1 sulfuric acid. Four individually cased 5-pint bottles of class 1 sulfuric acid packaged as specified in 5.1.1.1 shall be packed upright as specified in 5.2.1.1 except that the box shall conform to grade V3c of PPP-B-636.

5.2.2.2 One-gal quantity.

(a) Class 1 sulfuric acid. Four 1-gal containers of sulfuric acid packaged as specified in 5.1.1.2(a) shall be packed upright as specified for the four single bottle cases in 5.2.1.1 except that the box shall conform to type domestic, style A, B, or I, grade B of PPP-B-601 or class 1, style optional of PPP-B-585.

(b) Classes 2, 3, and 4 sulfuric acid. Four packages of class 2, 3, or 4 sulfuric acid prepared as specified in 5.1.2.1 shall be packaged upright in a snug-fitting box meeting the minimum requirements of grade V3c of PPP-B-636. Motion of contents shall be prevented by the insertion, where needed, of pads formed from fiberboard conforming to grade V3c of PPP-F-320. The box shall be closed and reinforced as specified in the appendix to PPP-B-636. The containers shall show no evidence of leakage of contents when tested as specified in 4.2.5.1.

5.2.2.3 Five-gal quantity, class 2 sulfuric acid. A quantity of 5 US gal (+6 or -0 oz) of class 2 sulfuric acid shall be packed as specified in 5.2.1.3 except that the type I, class 1 container of PPP-C-1337 or DOT Specification 34 as specified (see 6.2) shall be required.

5.2.2.4 Six and one-half-gal quantity, class 1 sulfuric acid. A quantity of 6-1/2 US gal (+8 or -0 oz) of class 1 sulfuric acid shall be packed as specified in 5.2.1.4 except that carboys conforming to DOT Specification 1D, 1X, or 34 as specified (see 6.2) shall also be permitted and reinforcement strapping shall not be required unless specified by DOT regulations.

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5.2.2.5 Fifteen-gal quantity, class 3 sulfuric acid. A quantity of 15 US gal (+1 or -0 pint) of class 3 sulfuric acid shall be packed as specified in 5.2.1.5 except that the container of PPP-C-1337, if specified, shall conform to type I, class 2 of PPP-C-1337.

5.2.3 Level C.

5.2.3.1 Civil agencies. Sulfuric acid shall be packed to assure carrier acceptance and safe delivery to the first domestic destination. Containers shall be in accordance with DOT regulations and applicable carrier regulations.

5.2.3.2 Military activities. Sulfuric acid shall be packed in accordance with MIL-STD-1188.

5.3 Marking.

5.3.1 Civil agencies. Shipments shall be marked in accordance with DOT regulations, Fed. Std. No. 123, and the precautionary marking specified in 5.3.3.

5.3.2 Military activities. Levels A and B shipments shall be marked in accordance with DOT regulations and MIL-STD-129. Level C shipments shall be marked in accordance with MIL-STD-1188. All packages and packs shall be marked to show lot number, date of manufacture of contents, and the precautionary marking specified in 5.3.3.

5.3.3 Precautionary marking. Each container shall be marked to clearly show the top of the container by the use of an arrow and the word "UP." Each fiberboard box specified in 5.1.1.2(b) and 5.1.2.1 and similar commercial containers shall show the directions and precautions to be observed for opening and pouring the contents; the marking shall include the words "DO NOT REMOVE LINER FROM CARTON" and an arrow and the word "POUR" indicating that the container should be held with the pouring spout in the uppermost position while the contents are being poured into a storage battery. Each container shall also be marked to show, as a minimum, the following:

DANGER! CAUSES SEVERE BURNS

Do not get in eyes, on skin, or on clothing.
Personal protective equipment, to include eye protection, must be worn when dispensing the contents of this container.

Adequate water supply for quick drenching or flushing the eyes and body must be available within the work area for emergency use.

In case of contact, immediately flush eyes and skin with generous quantities of water for at least 20 minutes; for eyes, get medical attention.

Do not add water to contents of this container because of violent reaction.

5.4 Palletization. Like packs of sulfuric acid shall be palletized upright together. Levels A and B packs shall be palletized in accordance with the applicable requirements of MIL-STD-147. Polyethylene containers with or without a fiberboard overpack and carboys inclosed in foamed polystyrene shall be palletized using the wood cap and inverted wood cap (storage aid 5), side framing (storage aid 10), and end framing (storage aid 12) of MIL-STD-147. The pallet shall conform to new, unused, seasoned softwood or hardwood, type IV of NN-P-71. Level C packs for military activities shall be palletized in accordance with the policies established in MIL-STD-1188. Level C packs for civil agencies shall be palletized to assure safe delivery to the first destination. Excess shipping containers which do not constitute a full pallet course and have a combined gross weight of 200 pounds or more shall be prepared for shipment in a manner which will assure transport of the containers in a unitized state from the initial source to the ultimate destination. The containers shall be consolidated in such a manner that will enable the load to be moved by forklift or other mechanical means. Excess shipping containers which do not constitute a full pallet course and have a combined gross weight of 199 pounds or less may be shipped as individual containers.

6. NOTES

6.1 Intended use. 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 should never be used undiluted in lead storage batteries.

6.2 Ordering data. Purchasers should select the preferred options permitted herein and include the following information in procurement documents:

- (a) Title, number, and date of this specification.
- (b) Class of sulfuric acid required (see 1.2).
- (c) Level of packaging required (see 5.1).

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- (d) Level of packing required (see 5.2).
- (e) If the four packages of class 2, 3, or 4 sulfuric acid prepared in 5.1.1.2(b) shall be packed in a box conforming to PPP-B-640 [see 5.2.1.2(b)].
- (f) Level A container required for 6-1/2 gal quantity of class 1 sulfuric acid (see 5.2.1.4).
- (g) Level A container required for 15-gal quantity of class 3 sulfuric acid (see 5.2.1.5).
- (h) Level B container required for 5-gal quantity of class 2 sulfuric acid (see 5.2.2.3).
- (i) Level B container required for 6-1/2-gal quantity of class 1 sulfuric acid (see 5.2.2.4).
- (j) Quantity required for level C packaging (see 5.1.3.1 and 5.1.3.2).

6.3 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.

6.4 Significant places. For the purpose 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 the Recommended Practice for Indicating Which Places of Figures Are To Be Considered Significant in Specified Limiting Values (ASTM E29).

MILITARY INTEREST:

Civil Agency Coordinating Activities:

Custodians:

EPA
GSA-FSS

Army - EA
Navy - SH
Air Force - 68

Preparing activity:

Army - EA

Review activities:

Army - MD
Navy - YD
DSA - GS

User activities:

Army - MI
Navy - MC, SA

U.S. GOVERNMENT PRINTING OFFICE : 1979 - 281-172/1237

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