

O-S-00801D (ARMY-EA)

July 10, 1990

INTERIM REVISION OF

O-S-801C

April 17, 1990

INTERIM FEDERAL SPECIFICATION**SULFURIC ACID, ELECTROLYTE; FOR STORAGE BATTERIES**

This interim Federal specification was developed by the U.S. Army Chemical Research, Development and Engineering Center, ATTN: SMCCR-PET-S, Aberdeen Proving Ground, MD 21010-5423, based upon currently available technical information. It is recommended that Federal agencies use it in procurement and forward recommendations for changes to the preparing activity at the address shown above.

The General Services Administration has authorized the use of this interim Federal specification as a valid exception to O-S-801C, dated April 17, 1990.

1. SCOPE

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

FSC 6810

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

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)
- NN-P-71 - Pallets, Material Handling, Wood, Stringer Construction, 2-Way and 4-Way (Partial)
- PPP-B-636 - Boxes, Shipping, Fiberboard
- PPP-C-96 - Cans, Metal, 28 Gauge and Lighter
- PPP-C-186 - Containers, Packaging and Packing for Drugs, Chemicals, and Pharmaceuticals
- PPP-T-66 - Tape, Packaging, Vinyl Plastic Film

Federal Standards:

- FED. TEST METHOD STD. NO. 101 - Test Procedures for Packaging Materials
- Fed. Std. No. 123 - Marking for Shipment (Civil Agencies)
- 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 Federal specifications, standards, and commercial item descriptions as outlined under General Information in the Index of Federal Specifications, Standards and Commercial Item Descriptions. The Index, which includes cumulative bimonthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

(Single copies of this specification, other Federal specifications, and commercial item descriptions required by activities outside the Federal Government for bidding purposes are available without charge from General Services Administration Business Service Centers in Boston, MA; New York, NY; Washington, DC; Philadelphia, PA; Atlanta, GA; Chicago, IL; Kansas City, MO; Fort Worth, TX; Denver, CO; San Francisco, CA; Los Angeles, CA; and Seattle, WA.

(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 Specifications:

MIL-C-5501/7 - Caps and Plugs, Protective, Dust and Moisture Seal (Cap-Plug, General Purpose)

Military Standards:

MIL-STD-129 - Marking for Shipment and Storage
MIL-STD-147 - Palletized Unit Loads

(Copies of military specifications and standards required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

Code of Federal Regulations (CFR):

29 CFR 1910.1200 - Occupational Safety and Health Standard on Hazard Communication
49 CFR 171 to 199 - Hazardous Materials Regulations

(The Code of Federal Regulations and the Federal Register (FR) are for sale on a subscription basis by 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.)

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

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, 1000 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, W1V 0AE, England.)

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ASTM Standards:

- C 516 - Vermiculite Loose Fill Thermal Insulation
D 1193 - Reagent Water

(Application for copies should be addressed to ASTM, 1916 Race Street, Philadelphia, PA 19103.)

(Non-Government standards and other publications are normally available from the organizations that prepare or distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.3 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

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

3.2 Color. Sulfuric acid, electrolyte 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, electrolyte 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, electrolyte shall conform to the maximum impurities characteristics of table II when tested as specified therein.

3.5 Material Safety Data Sheets. Material Safety Data Sheets for sulfuric acid, electrolyte shall be prepared and submitted by the contractor in accordance with FED-STD-313 (see 6.3).

TABLE II. Maximum impurities

| Impurity | 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 | 0.04 | 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

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 (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 Responsibility for compliance. 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 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 Contractor assurance of compliance. The contractor's quality program or detailed inspection system shall provide assurance of compliance of all characteristics with the applicable drawing, special packaging instruction, and specification requirements using, as a minimum, the conformance criteria specified herein.

4.1.3 Alternative inspection provisions. Alternative 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 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, 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 6.4).

4.2.2 Sampling.

4.2.2.1 For examination of packaging. Sampling shall be conducted in accordance with table III. The sample unit shall be one filled unit or shipping container, as applicable, ready for shipment.

TABLE III. Sampling for container examination and tests (except shock resistance)

| Number of containers in batch or lot | Number of sample containers |
|--------------------------------------|-----------------------------|
| 2 to 15 | 2 |
| 16 to 50 | 3 |
| 51 to 150 | 5 |
| 151 to 500 | 8 |
| 501 to 3,200 | 13 |
| 3,201 to 35,000 | 20 |
| 35,001 to 500,000 | 32 |
| Over 500,000 | 50 |

4.2.2.2 For sulfuric acid, electrolyte test. See 6.5 for sampling and testing precautions. Sampling shall be conducted in accordance with table IV. 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 IV. Sampling for sulfuric acid, electrolyte 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 |

4.2.2.3 For container leakage test. Sampling shall be conducted in accordance with table III. The sample unit shall be one 5-pint bottle of class 1, 2, 3, or 4 sulfuric acid, electrolyte or one 1-gallon container of class 1 sulfuric acid, electrolyte, as applicable.

4.2.2.4 For container contamination test. Sampling shall be conducted in accordance with table III. The sample unit shall be one 1-gallon bottle of class 2, 3, or 4 sulfuric acid, electrolyte, as applicable.

4.2.2.5 For container shock resistance test. Five sample units shall be selected. The sample unit shall be one filled and closed box containing a 1-gallon container of class 2, 3, or 4 sulfuric acid, electrolyte, as applicable.

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. Sample unit and shipping containers shall be examined for the characteristics listed below. Failure of any sample container to conform to all characteristics shall be cause for rejection of the lot represented.

- (a) Contents per container
- (b) Container
- (c) Container closure
- (d) Snug-fitting plug evident, correct, and properly seated (when required)
- (e) Spout evident and correct (when required)
- (f) Vermiculite evident and correct (when required)
- (g) Container free of damage and leaks
- (h) Marking evident, correct, and legible
- (i) Palletization (when required)

4.2.3.2 For sulfuric acid, electrolyte tests. Each sample specimen taken in 4.2.2.2 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.

4.2.3.3 For container leakage test. Each sample container taken in 4.2.2.3 shall be tested as specified in 4.2.5.1. Failure of the test by any container shall be cause for rejection of the lot represented.

4.2.3.4 For container contamination test. Each sample container taken in 4.2.2.4 shall be tested as specified in 4.2.5.2. Failure of the test by any container shall be cause for rejection of the lot represented.

4.2.3.5 For container shock resistance test. Each sample container taken in 4.2.2.5 shall be tested as specified in 4.2.5.3. Failure of the test by any container shall be cause for rejection of the lot represented.

4.2.4 Sulfuric acid, electrolyte tests. See 6.5 for sampling and testing precautions. Water 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:

4.2.4.1 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 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 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:

$$\text{Percent fixed residue} = \frac{100A}{BC}$$

where: A = Weight of residue in grams,

B = Milliliters of specimen, and

W = 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.

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 copper 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 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

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

$$\text{Percent zinc} = \frac{100A}{BC}$$

where: A = Weight of collected precipitate in grams,
 B = Milliliters of specimen, and
 W = Specific gravity of the 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.

(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 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 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, 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 reagent 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 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 reagent 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 and add, by pipet, 5 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 (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 $75^{\circ} \pm 5^{\circ}\text{F}$ for 4 hours. Observe the container for evidence of leakage during and at the end of the 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 $-40^{\circ} \pm 2^{\circ}\text{F}$ prior to testing. Subject the container to a free fall drop test in accordance with procedures B and E, method 5007.1 of FED. TEST METHOD STD. 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. PACKAGING

5.1 Packaging. Packaging shall be in accordance with the applicable requirements of 49 CFR 171 to 199 and the International Civil Aviation Organization - Technical Instructions for Safe Transport of Dangerous Goods by Air (ICAO-TDGA) or the International Maritime Organization - International Maritime Dangerous Goods Code (IMO-IMDGC), as applicable to the mode of transportation. The packaging shall meet the applicable packaging performance tests specified in ICAO-TDGA or IMO-IMDGC, as applicable.

5.2 Unit packing.

5.2.1 Five-pint (2.5-liter) quantity. A quantity of 5 pints (+5/8 or -0 fluid ounce) (2.5 liters) of sulfuric acid shall be unit packed in a nominal 5-pint (2.5-liter) screw-cap glass or polyethylene plastic bottle conforming to an IP.1 or IP.2 container of ICAO-TDGA or IMO-IMDGC, as applicable. The IP.1 container shall conform to group A, class 1, type d,

grade optional with closure A or R, and outer seal A of PPP-C-186. The material used for the bottle shall conform to type I, class M or H, grade 1 or 2 of L-P-390. The thickness of the bottle shall be not less than 0.030 inch. The bottle shall be designed with a finish adapted to a screw cap conforming to closure A or R of PPP-C-186. The closure of the bottle shall conform to closure A with a separate liner or closure R of PPP-C-186. The tightened screw cap shall be secured to the neck of the bottle by a strip of tape applied circumferentially around, and centered over, the juncture between the skirt of the cap and shoulder of the bottle neck. The tape shall have a nominal width of 2 inches and a length of not less than one and one-third times the circumference of the screw-cap closure. The tape shall conform to type 1, class optional of PPP-T-66. Alternately, a self-shrinking outer seal meeting the minimum performance requirements of PPP-T-66 tape shall be acceptable. There shall be no evidence of container leakage when the bottle is tested as specified in 4.2.5.1. Each bottle shall be placed upright and centered in a can with vermiculite cushioning. The can shall conform to type V, class 2, type VI tin or terneplate of PPP-C-96. The can shall be coated in accordance with plan B, with side seams striped of PPP-C-96. Sufficient vermiculite conforming to type I, grade 3 of ASTM C 516 shall be used in each can to assure absorption of the entire contents of the bottle in the event of leakage or breakage. The seams of the slip-cover can shall be sealed with the same kind of tape used for the bottle. The IP.2 container shall be the same as specified above except that the bottle shall conform to group A, class 2, style 1, grade 2, closure A, seal A of PPP-C-186.

5.2.2 One-gallon (4-liter) quantity, class 1. A quantity of 1 gallon (+1 or -0 ounce) (4 liters) of sulfuric acid shall be unit packed as specified in 5.2.1 except the container shall be a nominal 1-gallon (4-liter) bottle or jug.

5.2.3 One-gallon (4-liter) quantity, classes 2, 3, and 4. A quantity of 1 gallon (+1 or -0 ounce) (4 liters) of sulfuric acid shall be unit packed in a nominal 1 gallon (4-liter) rectangular polyethylene bottle conforming to the IP.2 container of ICAO-TDGA or IMO-IMDGC, as applicable, in a fiberboard box conforming to class domestic, type CF, variety SW, grade 200 of PPP-B-636. 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, 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. Sufficient vermiculite conforming to

type I, grade 3 of ASTM C 516 shall be used in each bottle to assure absorption of the entire contents of the bottle in the event of leakage or breakage. 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-B-636. The filled and closed box shall show no evidence of leakage when tested for shock resistance as specified in 4.2.5.3.

5.3 Packing. Packing shall be level A or B as specified (see 6.2).

5.3.1 Level A.

5.3.1.1 Five-pint (2.5-liter) quantity. Four 5-pint (2.5-liter) bottles unit packed as specified in 5.2.1 shall be packed upright in a close-fitting, water-repellent treated wooden or plywood box. The wooden box shall conform to the requirements of a 4C1 container of ICAO-TDGA or IMO-IMDGC, as applicable. The plywood box shall conform to the requirements of a 4D container of ICAO-TDGA or IMO-IMDGC, as applicable. In addition the plywood shall be not less than 3/8 inch thick and the cleats 3/4 inch thick by 1-3/4 inch wide. Each box shall be closed and reinforced as specified in accordance with the general packing requirements of ICAO-TDGA or IMO-IMDGC, as applicable.

5.3.1.2 One-gallon (4-liter) quantity. Four 1-gallon bottles of sulfuric acid unit packed as specified in 5.2.2 or 5.2.3 shall be packed as specified in 5.3.1.1.

5.3.1.3 Five-gallon (20-liter) quantity. A quantity of 5 gallons (+5 or -0 ounces) (20 liters) of sulfuric acid shall be packed in a nominal 5-gallon (20-liter) composite container conforming to the requirements of a 6HA1 container of ICAO-TDGA or IMO-IMDGC, as applicable. In addition, the outer metal body shall be a minimum 20 gauge and the plastic insert shall be a minimum of 0.015 inch thick.

5.3.1.4 Six and one-half-gallon (25-liter) quantity. A quantity of 6-1/2 gallons (+7 or -0 ounces) (25 liters) of sulfuric acid shall be packed in a nominal 6-1/2-gallon (25-liter) composite container or carboy conforming to the requirements of a 6HA2 or 6HD2 container of ICAO-TDGA or IMO-IMDGC, as applicable.

5.3.1.5 Fifteen-gallon (57-liter) quantity. A quantity of 15 gallons (+15 or -0 ounces) (57 liters) of sulfuric acid shall be packed as specified in 5.3.1.3 except the container shall be a nominal 15-gallon (57-liter) container.

5.3.2 Level B.

5.3.2.1 Five-pint (2.5-liter) quantity. Four 5-pint (2.5-liter) bottles of sulfuric acid unit packed as specified in 5.2.1 shall be packed upright in a close-fitting, weather-resistant fiberboard box conforming to the requirements of a 4G container of ICAO-TDGA or IMO-IMDGC, as applicable. Each box shall be closed and reinforced as specified in

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accordance with the general packing requirements of ICAO-TDGA or IMO-IMDGC, as applicable.

5.3.2.2 One-gallon (4-liter) quantity. Four 1-gallon (4-liter) containers of sulfuric acid shall be packed as specified in 5.3.2.1.

5.4 Marking.

5.4.1 Container compliance markings. Each shipping container shall be marked in accordance with ICAO-TDGA or IMO-IMDGC, as applicable.

5.4.2 Civil agencies. In addition to the marking required by 5.4.1, shipments shall be marked in accordance with Fed. Std. No. 123 and shall show the date of manufacture and lot or batch number of the sulfuric acid.

5.4.3 Military activities. In addition to the marking required by 5.4.1, level A and B shipments shall be marked in accordance with MIL-STD-129 and shall show the date of manufacture and lot or batch number of the sulfuric acid.

5.4.4 Hazard class label. Each shipping container and pallet load shall be labeled in accordance with 49 CFR 171 to 199 and either ICAO-TDGA or IMO-IMDGC, as applicable.

5.4.5 Proper shipping name. Each shipping container and pallet load shall be marked with the proper shipping name in accordance with 49 CFR 171 to 199 and either ICAO-TDGA or IMO-IMDGC, as applicable.

5.4.6 Precautionary marking. Each unit and shipping container shall be marked or labeled, as applicable, in accordance with the 29 CFR 1910.1200(f) to show the required precautionary information. Each outer container shall be marked to show the top of the container by use of an arrow and the word "UP". Each fiberboard box specified in 5.2.3 and 5.3.2.2 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 plus 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.

5.4.7 Shelf life marking. Each plastic container shall be marked in accordance with MIL-STD-129 to indicate a 12-month shelf life.

5.5 Palletization. Like packs of sulfuric acid shall be palletized upright together. All shipments of containers of 5 gallons or less shall conform to load type VI in accordance with MIL-STD-147 and the pallet shall conform to type IV or V of NN-P-71. When

specified, all other shipments of sulfuric acid shall be palletized in accordance with the applicable requirements of MIL-STD-147 (see 6.2).

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

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, electrolyte should never be used undiluted in lead storage batteries.

6.2 Acquisition requirements. 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 quantity required
- (d) Level of packing required (see 5.3)
- (e) If palletization is required for shipments of containers over 5 gallons (see 5.5)

6.3 Material Safety Data Sheets. Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313. The pertinent mailing addresses for submission of data are listed in FED-STD-313.

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 Sampling and testing precautions. This specification requires inspection of chemical material which 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 (type 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 Submission of alternative inspection provisions. Proposed alternative inspection provisions should be submitted by the contractor to the procuring contracting officer for

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evaluation and approval by the technical activity responsible for preparation of this specification.

6.7 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 ASTM E 29.

6.8 Subject term (key word) listing.

- Acid content
- Ammonium
- Antimony
- Arsenic
- Chloride
- Contamination
- Copper
- Fixed residue
- Iron
- Leakage
- Material Safety Data Sheet
- Manganese
- Nickel
- Nitrate
- Organic matter
- Platinum
- Selenium
- Shock resistance
- Specific gravity
- Sulfurous acid
- Zinc

Custodian:

Army - EA

Preparing activity:

Army - EA

Project No. 6810-A067