O-O-670B

July 19, 1985

SUPERSEDING

O-O-670a

January 9, 1968

### FEDERAL SPECIFICATION

URTHOPHOSPHORIC (PHOSPHORIC) ACID, TECHNICAL

This specification is approved by the Assistant Administrator, Office of Federal Supply and Services, General Services Administration, for the use of all Federal agencies.

## 1. SCOPE

- 1.1 Scope. This specification covers two classes of technical grade orthophosphoric acid, commonly called phosphoric acid.
- 1.2 Classification. Orthophosphoric acid shall be of the following classes as specified (see 6.2):

Class 1 - 85 percent acid Class 2 - 75 percent acid

## 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:

NN-P-71 - Pallets, Material Handling, Wood, Stringer Construction,
2-Way and 4-Way (Partial)

PPP-B-585 - Boxes, Wood, Wirebound

PPP-B-636 - Boxes, Shipping, Fiberboard

PPP-C-186 - Containers, Packaging and Packing for Drugs, Chemicals, and
Pharmaceuticals

PPP-C-2020 - Chemicals, Liquid, Dry, and Paste: Packaging Of

PPP-F-320 - Fiberboard; Corrugated and Solid, Sheet Stock (Container

Grade) and Cut Shapes

## Federal Standard:

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

**FSC 6810** 

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# 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

(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.)

# Code of Federal Regulations (CFR)

49 CFR 171 to 179 - 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.

## Reagent Chemicals, Sixth Edition:

(Application for copies should be addressed to the American Chemical Society, 1155 Sixteenth Street, NW, Washington, DC 20036.)

# ASTM Standards:

D 1193 - Reagent Water

E 29 - Indicating Which Places of Figures are to be Considered Significant in Specified Limiting Values

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

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein (except for the Code of Federal Regulations), the text of this specification shall take precedence.

## 3. REQUIREMENTS

- 3.1 Appearance and odor. Orthophosphoric acid shall be a clear, water-white liquid with no foreign odor, shall be free from sediment and suspended matter, and shall not be separated into two or more liquid layers when tested as specified in 4.2.4.1.
- 3.2 Physical and chemical characteristics. Orthophosphoric acid shall conform to the applicable physical and chemical characteristics of table I when tested as specified therein.

Class 1 Class 2 Test Characteristic\* Minimum : Maximum : Minimum : Maximum : paragraph Total acid content (as HaPO4) 85.0 75.0 4.2.4.2 Sulfates (as SO<sub>h</sub>) 0.35 : 4.2.4.3 Arsenic (As) 0.001: 0.06 4.2.4.4 Alkali and other phos-4.2.4.5 phates 0.25 Heavy metals (as Pb) 0.005 4.2.4.6 Volatile acids (as acetic acid) 0.10 4.2.4.7 Specific gravity 20°/4°C 1.690 1.580 4.2.4.8

TABLE I. Physical and chemical characteristics

<sup>\*</sup> All values are percent by weight except specific gravity.

### 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 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 the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

# 4.2 Quality conformance inspection.

4.2.1 <u>Lotting</u>. A lot shall consist of the orthophosphoric acid of one class, produced by one manufacturer, at one plant, 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 examination of packaging. Sampling shall be conducted in accordance with MIL-STD-105, inspection level S-2.

4.2.2.2 For orthophosphoric acid test. See 6.4 for sampling and testing precautions. Sampling shall be conducted in accordance with table II. A representative specimen of approximately 500 grams (g) 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 II.	Sampling	for	orthophosphoric	acid	test

Number of containers in batch of	or lot : Num	ber of sample conta	ainers
	:		
2 to 25	:	2	
26 to 150	:	3	
151 to 1,200	•	5	
1,201 to 7,000	:	8	
7,001 to 20,000	:	10	
Over 20,000	:	20	
	:		

4.2.2.3 For container leakage test. Sampling shall be conducted in accordance with MIL-STD-105, inspection level S-3.

# 4.2.3 Inspection procedure.

- 4.2.3.1 For examination of packaging. 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 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
  - (d) Container damaged or leaking
  - (e) Fiberboard liners, partitions, or pads missing or not as specified (when required)
  - (f) Marking incorrect, missing or illegible
  - (g) Unitization not as specified
- 4.2.3.2 For orthophosphoric acid test. See 6.4 for sampling and testing precautions. 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. The sample unit shall be one filled container. The sample containers selected in 4.2.2.3 shall be tested as specified in 4.2.5 using an AQL of 1.5 percent defective.
- 4.2.3.4 Significant places. For the purpose of determining conformance with this specification, an observed or calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM E 29.
- 4.2.4 Orthophosphoric acid tests. 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. Tests shall be conducted as follows:
- 4.2.4.1 Appearance and odor. Pour approximately 25 milliliters (mL) of the thoroughly mixed specimen into a clean, dry test tube and examine for odor. Allow the specimen to stand for at least 10 minutes and then examine visually for clearness and color and for sediment, suspended matter, and separated liquid layers.
- 4.2.4.2 Total acid content. Weigh to the nearest 0.01 g appproximately 1 mL of the specimen into a 250-mL Erlenmeyer flask and dilute with 120 mL of water. Ada 0.5 mL of 0.1-percent thymolphthalein solution as the indicator and titrate with approximately 1N sodium hydroxide solution which has been standardized. Calculate the percent by weight of orthophosphoric acid as follows:

Percent orthophosphoric acid = 4.9AB

where: A = Milliliters of sodium hydroxide solution used,

B = Normality of sodium hydroxide solution, and

W = Weight of specimen in grams.

4.2.4.3 <u>Sulfates</u>. Weigh to the nearest 0.01 g approximately 10 g of the specimen into a 1000-mL beaker. Dilute with 500 mL of water, add 15 mL of concentrated hydrochloric acid, heat to boiling, and add 25 mL of 10-percent barium chloride solution. Boil for an additional 30 minutes, digest on a steam bath for 2 hours, and allow to stand overnight. Filter the precipitate through a tared filter crucible weighed to the nearest 0.1 milligram (mg), wash with hot water, ignite, cool, and weigh to the nearest 0.1 mg. Calculate percent by weight of sulfate acid as follows:

Percent sulfate = 
$$\frac{41.2 (A - B)}{W}$$

where: A = Weight of crucible and precipitate in grams,

B = Weight of crucible in grams, and

W = Weight of specimen in grams.

# 4.2.4.4 Arsenic.

- (a) Class 1 orthophosphoric acid. Weigh 3.0 g of the specimen and dilute to 30 mL with water. Test a 3-mL aliquot of the solution for arsenic by the colorimetric comparative procedure for arsenic found in Reagent Chemicals, Sixth Edition. For the standard use 0.003 mg of arsenic.
- (b) Class 2 orthophosphoric acid. Weigh 1.0 g of the specimen and dilute to 600 mL with water. Test a 3-mL aliquot of the solution for arsenic by the colorimetric comparative procedure for arsenic found in Reagent Chemicals, Sixth Edition. For the standard use 0.003 mg of arsenic.
- 4.2.4.5 Alkali and other phosphates. Weigh to the nearest 0.01 g approximately 5 g of the specimen into a 250-mL Erlenmeyer flask. Add 100 mL of 95-percent ethyl alcohol. Stopper, and allow to stand for 1 hour at 20° to 30°C. Filter through a tared, clean, dry filter crucible weighed to the nearest 0.1 mg and wash with 95-percent ethyl alcohol until the filtrate no longer shows acid with moistened blue litmus paper. Do not allow the filter to run dry during the filtering and washing operations. Dry the filter crucible and residue to constant weight at 100° to 105°C, cool, and weigh to the nearest 0.1 mg. Calculate the percent by weight of alkali and other phosphates as follows:

Percent alkali and other phosphates = 
$$\frac{100 \text{ (A - B)}}{\text{W}}$$

where: A = Weight of filter crucible and residue in grams,

B = Weight of filter crucible in grams, and

W = Weight of specimen in grams.

4.2.4.6 Heavy metals. Dilute 3 mL of the specimen with water and make up to a volume of 150 mL. Neutralize 30 mL of this solution with ammonium hydroxide solution using litmus paper as the indicator. Add 1 mL of concentrated hydrochloric acid and 5 mL of a saturated solution of hydrogen sulfide made by passing hydrogen sulfide gas into cold water, and dilute to 100 mL. Any color produced shall be no darker than that produced when 3 mL of lead nitrate solution (2.6 mg per liter) is tested in the same manner as the 30 mL of the specimen solution.

4.2.4.7 Volatile acids. Weigh to the nearest 0.1 g approximately 40 g of the specimen and dilute with 75 mL of carbon dioxide-free water. Distill off 50 mL of the solution. Titrate the distillate with approximately 0.1N sodium hydroxide solution which has been standardized, using three drops of phenolphthalein indicator solution. Calculate the percent by weight of volatile acids, as acetic acid, as follows:

Percent volatile acids =  $\frac{6AB}{V}$ 

where: A = Milliliters of sodium hydroxide solution used,

B = Normality of sodium hydroxide solution, and

W = Weight of specimen in grams.

4.2.4.8 Specific gravity. Determine the specific gravity at 20°/4°C by means of a pycnometer or other suitable method.

- 4.2.5 Container leakage test. Place the filled container in each of the following positions, as applicable, and leave it in each for a period of 15 minutes.
  - (a) Upright
  - (b) Upside down
  - (c) On one side (or one quadrant)
  - (d) On one end (or second quadrant)
  - (e) On other side (or fourth quadrant)

Examine the container after each period for any evidence of leakage.

# 5. PACKAGING

5.1 Preservation. Orthophosphoric acid shall be preserved level A, B or C as specified (see 6.2) in accordance with Department of Transportation (DOT) regulations, any other regulations applicable to the intended mode of transportation, and the general requirements of PPP-C-2020.

# 5.1.1 Level A.

5.1.1.1 Unit packing. Orthophosphoric acid shall be unit packed level A in an 8- or 200-pound (1b) quantity as specified (see 6.2).

- (a) Eight-1b quantity. A quantity of 8 (+0.1 or -0) 1b of orthophosphoric acid shall be unit packed in a glass bottle of minimum size to accommodate the acid, and conforming to group A, class 1, type of glass optional, style 1 narrow mouth, grade optional of light penetration, closure A or R, and outer seal A of PPP-C-186. The bottle shall be cushioned, closure end uppermost, in enclosure B of PPP-C-2020. There shall be no leakage of contents from the bottle when tested prior to application of the outer seal A and as specified in 4.2.5.
- (b) Two hundred-lb quantity. A quantity of 200 (+2.0 or -0) lb of orthophosphoric acid shall be unit packed in a nominal 15-gallon (gal) capacity polyethylene drum conforming to DOT Specification 34. The drum shall be closed in accordance with the drum manufacturer's instructions. There shall be no evidence of leakage when the container is tested as specified in 4.2.5.
- 5.1.1.2 <u>Intermediate packing</u>. Four unit packs of the 8-lb quantity, unit packed as specified in 5.1.1.1(a), shall be intermediately packed with closure ends uppermost in a close-fitting fiberboard box conforming to grade V3c of PPP-B-636 and furnished with close-fitting cells. The cells shall be formed by use of half-slotted fiberboard partitions of full-can height. Motion of contents shall be prevented by inserting fiberboard pads as needed. The box shall be closed as specified for closure of boxes intended for use as interior containers in PPP-B-636.

# 5.1.2 <u>Level B</u>.

- 5.1.2.1 Unit packing. Orthophosphoric acid shall be unit packed level B in an 8- or 200-lb quantity as specified (see 6.2).
- (a) <u>Fight-1b quantity</u>. A quantity of 8 (+0.1 or -0) 1b of orthophosphoric acid shall be unit packed in a glass bottle as specified in 5.1.1.1(a), or a polyethylene bottle conforming to group A, class 2 plastic, style 1 narrow mouth, light penetration grade optional, closure A or R, outer seal A of PPP-C-186. Each bottle shall be further protected and cushioned with closure end uppermost, using an individual expanded polystyrene enclosure as specified for a DOT Specification 33A composite container. There shall be no evidence of leakage when the container is tested as specified in 4.2.5.
- (b) Two hundred-lb quantity. A quantity of 200 (+2.0 or -0) lb of orthophosphoric acid shall be unit packed level B in the same manner as specified for level A in 5.1.1.1.
- 5.1.2.2 Intermediate packing. Four unit packs of the 8-lb quantity, unit packed as specified in 5.1.2.1(a), shall be intermediately packed with closure ends uppermost in a close-fitting fiberboard box conforming to grade 275, type CF, class domestic of PPP-B-636. The box ends and sides shall be fully lined with fiberboard having flutes parallel to the vertical dimension of the box. Motion of contents shall be prevented by inserting fiberboard pads where needed. Liner and pads shall be formed from fiberboard conforming to grade 275, type CF, class domestic of PPP-F-320. The box shall be closed as specified in PPP-B-636 for the closure of boxes intended for use as interior containers.

## 5.1.3 Level C.

- 5.1.3.1 Unit packing. Orthophosphoric acid shall be unit packed level C in an 8- or 200-lb quantity as specified (see 6.2).
- (a) Eight-1b quantity. A quantity of 8 (+0.1 or -0) 1b of orthophosphoric acid shall be unit packed level C in a plastic bottle of minimum
  size to contain the contents in accordance with DOT regulations. The bottle
  shall conform to group A, class 2 plastic, style 1 narrow mouth, light penetration grade optional, closure A or R, outer seal A of PPP-C-186. The bottle
  shall be closed in accordance with the bottle manufacturer's instructions.
  There shall be no leakage of contents when the container is tested as specified
  in 4.2.5.
- (b) Two hundred-lb quantity. A quantity of 200 (+2 or -0) lb of orthophosphoric acid shall be unit packed level C in a polyethylene drum of minimum capacity to contain the contents. The drum shall conform to DOT Specification 34 and shall be closed in accordance with the drum manufacturer's instructions. There shall be no evidence of leakage of contents when the container is tested as specified in 4.2.5.
- 5.1.3.2 Intermediate packing. Four unit packs of orthophosphoric acid prepared as specified in 5.1.3.1(a) shall be intermediately packed in a close-fitting fiberboard box conforming to DOT Specification 12B. Each unit pack shall be placed with closure end uppermost in a close-fitting cell formed from full-bottle-height half-slotted fiberboard partitions made of fiberboard conforming to the same fiberboard as used for the box. The box shall be closed as specified for the closure of DOT Specification 12B boxes.
- 5.2 Packing. Orthophosphoric acid shall be packed level A, B or C as specified (see 6.2) in accordance with DOT regulations, any other regulations applicable to the intended mode of transportation, and the general requirements of PPP-C-2020.

## 5.2.1 Level A.

- 5.2.1.1 <u>Eight-lb quantity</u>. Two intermediate packs of the 8-lb quantity shall be packed side-by-side, with closure ends uppermost in a close-fitting wirebound box conforming to class 3, style optional of PPP-B-585. Fiberboard pads formed from material conforming to grade W5c of PPP-F-320 shall be inserted where needed to prevent motion of contents. The box shall be closed as specified in PPP-B-585.
- 5.2.1.2 Two hundred-lb quantity. The 200-lb quantity of orthophosphoric acid, unit packed as specified in 5.1.1.1(b), shall require no further protection for shipment other than unitization.

# 5.2.2 Level B.

- 5.2.2.1 <u>Eight-lb quantity</u>. Two intermediate packs of the 8-lb quantity of orthophosphoric acid shall be packed level B in the same manner as specified above for level A, except that the box shall conform to grade V3c of PPP-B-636, and shall be closed as specified therein for the closure of boxes intended for use as exterior shipping containers.
- 5.2.2.2 Two hundred-lb quantity. The 200-lb quantity, unit packed as specified in 5.1.2.1(b), shall require no further protection for shipment other than unitization.

# 5.2.3 <u>Level C</u>.

- 5.2.3.1 <u>Eight-lb quantity</u>. Two intermediate packs of the 8-lb quantity of orthophosphoric acid shall be packed level C in the same manner as specified for level A, except that the box shall conform to DOT Specification 12B.
- 5.2.3.2 Two hundred-lb quantity. The 200-lb quantity of orthophosphoric acid, unit packed as specified in 5.1.3.1(b), shall require no further protection for shipment other than unitization.
- 5.3 Unitization. Uniform quantities of level A, B, or C packs shall be palletized in accordance with the applicable requirements of MIL-STD-147 using load type I for boxes and load type III or IV, as applicable, for drums or pails. Pallets shall conform to the soft-wood pallet of NN-P-71, using the preserved pallet for all level A shipments.

### 5.4 Marking.

- 5.4.1 Civil agencies. Marking shall be in accordance with Fed. Std. No. 123.
- 5.4.2 <u>Military activities</u>. Containers and pallet loads shall be marked in accordance with DOT regulations and MIL-STD-129. Containers shall be marked to show date of manufacture and lot number of the orthophosphoric acid.
- 5.4.3 Precautionary marking. Each unit container of orthophosphoric acid shall be durably and legibly marked as follows:

#### HAZARD:

Corrosive, Eye and Skin.

#### DANGER! CAUSES SEVERE BURNS

Do not get in eyes, on skin, on clothing. Avoid breathing mist. Keep container closed.

Use with adequate ventilation. Wash thoroughly after handling.

FIRST AID: In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Call a physician.

## 6. NOTES

- 6.1 Intended use. Orthophosphoric acid is intended for use in the manufacture of metals, chemicals, and fertilizers, and in the cleaning of a variety of materials.
  - 6.2 Ordering data. Acquisition documents should specify the following:

    - (a) Title, number, and date of this specification,(b) Class of orthophosphoric acid required (see 1.2),
    - (c) Level of preservation and packing required (see 5.1 and 5.2), and
    - (d) Quantity to be unit packed level A, B, or C (see 5.1.1.1, 5.1.2.1, and 5.1.3.1).
- 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 Sampling and testing precautions. This specification requires inspection of chemical material which is potentially hazardous to personnel. Orthophosphoric acid is corrosive to eyes and skin. All applicable safety rules, regulations, and procedures must be followed in the handling and processing of this material.

MILITARY INTERESTS:

CIVIL AGENCY COORDINATING ACTIVITIES:

Custodians:

AGR-AMS COM-NBS GSA-7FCE

Army - EA

Navy - OS Air Force - 68

Preparing activity:

Review activities:

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