

INCH-POUND

O-O-690C

May 14, 1992

SUPERSEDING

O-O-690B

February 29, 1984

FEDERAL SPECIFICATION
OXALIC ACID, DIHYDRATE, TECHNICAL

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

1. SCOPE

1.1 Scope. This specification covers three classes of technical grade oxalic acid, dihydrate (hereinafter referred to as oxalic acid).

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

- Class 1 - Large crystals
- Class 2 - Small crystals
- Class 3 - Powder

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:

- PPP-B-585 - Boxes, Wood, Wirebound
- PPP-B-636 - Boxes, Shipping, Fiberboard

Comments or suggestions pertaining to this specification should be addressed to:
Commander, U.S. Army Chemical Research, Development and Engineering Center,
ATTN: SMCCR-PET-S, Aberdeen Proving Ground, MD 21010-5423.

AMSC N/A

FSC 6810

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

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- PPP-C-186 - Containers, Packaging and Packing for Drugs, Chemicals, and Pharmaceuticals
- PPP-F-320 - Fiberboard: Corrugated and Solid, Sheet Stock (Container Grade) and Cut Shapes

Federal Standards:

- 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 Government Activities

(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 Auburn, 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-P-15011 - Pallets, Material Handling, Wood Post Construction, 4-Way Entry

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 1900.1200 - Hazard Communication
- 49 CFR 171 to 199 - Hazardous Materials Regulations

(The Code of Federal Regulations and 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.

ASTM Standards:

- D 1193 - Reagent Water
- E 11 - Wire-Cloth Sieves for Testing Purposes

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

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.)

(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.

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3. REQUIREMENTS

3.1 Assay. Oxalic acid shall have a minimum assay of 99.0 percent by weight as $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ when tested as specified in 4.2.4.1.

3.2 Ash. Oxalic acid shall have a maximum ash content of 0.20 percent by weight when tested as specified in 4.2.4.2.

3.3 Particle size. Oxalic acid shall conform to the applicable particle size characteristics of table I when tested as specified in 4.2.4.3.

TABLE I. Particle size characteristics

Characteristic	Percent by weight		
	Class 1	Class 2	Class 3
Passing through a 2.00-millimeter sieve	85 min	100 min	None 20 min
Retained on a 1.40-millimeter sieve			
Retained on a 500-micrometer sieve		75 min	
Passing through a 75-micrometer sieve			

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

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 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 6.4.) 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 oxalic 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 6.5).

4.2.2 Sampling.

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

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TABLE II. Sampling for packaging examination and test

Number of containers in batch or lot	Number of sample containers
1 or 2	all
3 to 25	3
26 to 50	5
51 to 90	6
91 to 150	7
151 to 280	10
281 to 500	11
501 to 1,200	15
1,201 to 3,200	18
3,201 to 10,000	22
over 10,000	29

4.2.2.2 For oxalic acid test. See 6.6 for sampling and testing precautions. Sampling shall be conducted in accordance with table III. A representative specimen of approximately 200 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 III. Sampling for oxalic acid test

Number of containers in batch or lot	Number of sample containers
1 or 2	all
3 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 table II. The sample unit shall be one container.

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. Sample unit packs and packing containers shall be examined for the characteristics listed below. Failure of any sample unit pack or packing 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) Container free of damage and leaks
- (e) Fiberboard pads or partitions evident and correct, or as specified (when required)
- (f) Marking evident, correct, and legible
- (g) Unitization

4.2.3.2 For oxalic acid test. See 6.6 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 containers selected in 4.2.2.3 shall be tested as specified in 4.2.5. Failure of any test by any container shall be cause for rejection of the lot represented.

4.2.4 Oxalic 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 Assay (as $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$). Weigh to the nearest 0.1 milligram (mg) approximately 0.25 g of the specimen and completely dissolve in 100 milliliters (mL) of water. Add 15 mL of 1 to 1 sulfuric acid, heat to $65^\circ \pm 10^\circ\text{C}$ and titrate with 0.1 N potassium permanganate solution. Calculate the percent by weight oxalic acid, dihydrate as follows:

$$\text{Percent oxalic acid, dihydrate} = \frac{6.304 AB}{W}$$

where A = Milliliters of potassium permanganate solution used,
 B = Normality of potassium permanganate solution,
 W = Weight of specimen, in g.

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4.2.4.2 Ash. Weigh to the nearest 0.1 mg approximately 4 g of the specimen into a tared porcelain crucible. Burn the combustible matter off slowly and then ignite to constant weight. Cool in a desiccator and weigh. Calculate the percent by weight ash as follows:

$$\text{Percent ash} = \frac{100 (A - B)}{W}$$

where A = Weight of crucible and ignited residue, in g,
 B = Weight of crucible, in g,
 W = Weight of specimen, in g.

4.2.4.3 Particle size. Sieves shall conform to ASTM E 11. Weigh the sieves required and bottom pan. Where two sieves are specified, nest them with the coarser one on top. Weigh to the nearest 0.01 g approximately 100 g of the specimen and transfer to the top sieve and cover. Shake the sieve until the weight of the residue retained is not reduced by more than 0.1 g by further shaking for 1 minute. Calculate the percent by weight passing or retained.

4.2.5 Container leakage test. Place the container in each of the following positions, 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

Note: The metric equivalents given for inch-pound quantities are nominal values provided for informational purposes and should not be considered as quantity requirements.

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 the 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 One-pound (0.45-kilogram) quantity. A quantity of 1 pound (+1/8 or -0 ounces) (0.45 kilograms) of oxalic acid shall be unit packed in a 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, type optional glass, with screw-cap A or R and outer seal A of PPP-C-186. The IP.2 container shall conform to style 2 or 4, light penetration grade optional, with screw-cap A or R and outer seal A of PPP-C-186. The bottle shall be closed tightly in accordance with the closure manufacturer's instructions.

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

5.3.1 Level A.

5.3.1.1 One hundred-pound (45.36-kilogram) quantity. A quantity of 100 pounds (+1 or -0 ounces) (45.36 kilograms) of oxalic acid shall be packed in a nominal 15-gallon (60-liter) removable-head steel drum conforming to the requirements of a 1A2 container of ICAO-TDGA or IMO-IMDGC, as applicable. The container shall be furnished with a polyethylene bag liner and shall be constructed of minimum 14 gauge steel sheet.

5.3.2 Level B.

5.3.2.1 One-pound (0.45-kilogram) quantity. Twelve 1-pound (0.45-kilogram) bottles of oxalic acid, unit packed as specified in 5.2.1, shall be packed upright, in three rows of four bottles 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 bottle shall be placed in a close-fitting cell formed by full-bottle-height half-slotted fiberboard partitions. Motion of contents shall be prevented by inserting fiberboard pads where needed. Fiberboard pads and partitions shall be formed from material conforming to class weather-resistant, grade W5c of PPP-F-320. Each box shall be closed and reinforced as specified in accordance with the general packing requirements of ICAO-TDGA or IMO-IMDGC, as applicable.

5.4 Overpacking. Overpacking shall be level A or B as specified (see 6.2).

5.4.1 Level A.

5.4.1.1 One-pound (0.45-kilogram) quantity. Four 1-pound (0.45-kilogram) packs shall be overpacked in a close-fitting wirebound wood box. The wood box shall conform to class 3, style optional, with veneer face boards of PPP-B-585. Motion of contents in the box shall be prevented by inserting fiberboard pads, where needed, formed from material conforming to class weather-resistant, grade V3c of PPP-F-320. The box shall be closed as specified in the appendix to PPP-B-585.

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5.4.2 Level B.

5.4.2.1 One-pound (0.45-kilogram) quantity. Four 1-pound (0.45-kilogram) packs shall be overpacked level B in the same manner as specified for level A, except that the box shall conform to class weather-resistant, grade V3c of PPP-B-636. The fiberboard box shall be closed as specified in the appendix to PPP-B-636 for closure of boxes used as exterior containers.

5.5 Marking. Shipments for civil agencies shall be marked in accordance with Fed. Std. No. 123. Shipments for military activities shall be marked in accordance with MIL-STD-129.

5.5.1 Container compliance markings. Each shipping container shall be marked in accordance with 49 CFR 171 to 179 and either ICAO-TDGA or IMO-IMDGC, as applicable.

5.5.2 Hazard class label. Each shipping container and pallet load shall be labeled in accordance with 49 CFR 171 to 179 and either ICAO-TDGA or IMO-IMDGC, as applicable.

5.5.3 Proper shipping name. Each shipping container and pallet load shall be marked with the proper shipping name ("Corrosive solids, poisonous, n.o.s.") in accordance with 49 CFR 171 to 179 and either ICAO-TDGA or IMO-IMDGC, as applicable.

5.5.4 Precautionary markings. Each unit pack, pack, and shipping container shall be marked or labeled, as applicable, in accordance with 29 CFR, Hazards Communication, 1900.1200(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".

5.5.5 Overpack markings. Each overpack shall be marked "Inner packages comply with prescribed specification 4G."

5.6 Palletization. Uniform quantities of the same size packs of oxalic acid shall be palletized in accordance with MIL-STD-147, with pallet, for both level A and B shipments, conforming to type I of MIL-P-15011. Boxes of the 1-pound (0.45-kilogram) quantity shall be palletized using requirements for load type I of MIL-STD-147. Drums of the 100-pound (45.36-kilogram) quantity shall be palletized using requirements for load type III of MIL-STD-147.

6. NOTES

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

6.1 Intended use. Oxalic acid is intended for use as a laundry sour and as a floor bleach.

6.2 Acquisition requirements. Acquisition documents must specify the following:

- (a) Title, number, and date of this specification
- (b) Class of oxalic acid required (see 1.2)
- (c) Level of unit packing and packing required (see 5.2 and 5.3)
- (d) Level of overpacking required (see 5.4)
- (e) If palletization is required for shipment (see 5.6).

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 submissions of data are listed in FED-STD-313.

6.4 Submission of alternative inspection provisions. Proposed alternative inspection provisions should be submitted by the contractor to the procuring contracting officer for evaluation and approval by the technical activity responsible for preparation of this specification.

6.5 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.

6.6 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. 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.

6.7 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.

6.8 Changes from previous issues. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

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6.9 Subject term (key word) listing.

bleach, floor
laundry sour

MILITARY INTERESTS:

Custodians:

Army - EA
Air Force - 68

Review activities:

Army - MD
DLA - GS

CIVIL AGENCY COORDINATING ACTIVITIES:

GSA-FSS (9FTE-10)
HHS-NIH

Preparing activity:

Army - EA

Project No. 6810-1273

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.
2. The submitter of this form must complete blocks 4, 5, 6, and 7.
3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements.

I RECOMMEND A CHANGE:

1. DOCUMENT NUMBER

0-0-690C

2. DOCUMENT DATE (YYMMDD)

920514

3. DOCUMENT TITLE

OXALIC ACID, DIHYDRATE, TECHNICAL

4. NATURE OF CHANGE (Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)

5. REASON FOR RECOMMENDATION

6. SUBMITTER

a. NAME (Last, First, Middle Initial)

b. ORGANIZATION

c. ADDRESS (Include Zip Code)

d. TELEPHONE (Include Area Code)

(1) Commercial

(2) AUTOVON

(If applicable)

7. DATE SUBMITTED

(YYMMDD)

8. PREPARING ACTIVITY

a. NAME

U.S. Army Chemical Research, Development
and Engineering Center

b. TELEPHONE (Include Area Code)

(1) Commercial

(410) 671-3259

(2) AUTOVON

DSN 584-3259

c. ADDRESS (Include Zip Code)

Cdr, U.S. Army CRDEC

ATTN: SMCCR-PET-S

Aberdeen Proving Ground, MD 21010-5423

IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT:

Defense Quality and Standardization Office

5203 Leesburg Pike, Suite 1403, Falls Church, VA 22041-3466

Telephone (703) 756-2340 AUTOVON 289-2340