

JAN-A-465

18 APRIL 1947

JOINT ARMY-NAVY SPECIFICATION

ACID, ACETIC (FOR ORDNANCE USE)

This specification was approved by the War Department and the Navy Department for use of procurement services of the Army and Navy.

A. APPLICABLE SPECIFICATIONS AND OTHER PUBLICATIONS

A-1. *Specifications.*—The following specifications, of the issue in effect on date of invitation for bids, form a part of this specification:

U. S. ARMY SPECIFICATIONS

50-0-1—General Specification for Ammunition except Small Arms Ammunition.¹

100-2 —Standard Specification for Marking Shipments by Contractors.¹

NAVY DEPARTMENT SPECIFICATION

General Specifications for Inspection of Material.²

A-2. *Other publications.*—The following publications, of the issue in effect on date of invitation for bids, form a part of this specification:

BUREAU OF SUPPLIES AND ACCOUNTS PUBLICATION
Navy Shipment Marking Handbook.²

INTERSTATE COMMERCE COMMISSION REGULATIONS
Regulations for Transportation of Explosives and Other Dangerous Articles, etc.

B. GRADE

B-1. This specification covers one grade of acetic acid as hereinafter specified.

C. MATERIAL AND WORKMANSHIP

C-1. See section E.

D. GENERAL REQUIREMENTS

D-1. See section E.

E. DETAIL REQUIREMENTS

E-1. *Appearance.*—

E-1a. *Color.*—The sample shall show no more color than the standard containing 10 p.p.m. of platinum. (See par. F-4a(1).)

E-1b. *Suspended matter.*—None. (See par. F-4a(2).)

E-2. *Purity.*—Minimum, 99.90 percent.

E-3. *Freezing point.*—Minimum, 16.4°C.

¹ Applicable only to Army purchases.

² Applicable only to Navy purchases.

E-4. *Chlorides*.—None. (See par. F-4d.)

E-5. *Sulfates*.—None. (See par. F-4e.)

E-6. *Formic and sulfurous acids*.—None. (See par. F-4f.)

E-7. *Heavy metals*.—When tested in accordance with the methods described in paragraph F-4g, the absence of antimony, arsenic, bismuth, cadmium, cobalt, copper, lead, manganese, mercury, nickel, tin, and zinc shall be established.

F. METHODS OF SAMPLING, INSPECTION, AND TESTS

F-1. *Size of lot*.—Maximum, 100,000 pounds.

F-2. *Sampling*.—Select 10 percent, but in no case more than 10 containers of the containers comprising the lot so as to be representative of the entire lot. From each selected container remove sufficient material by means of a thief to form a composite sample of approximately 1 quart when the portions removed from the selected containers are united. Fill a 16-ounce glass-stoppered bottle with the composite sample. If the material is shipped in tank cars, remove the sample (approx. 1 quart) by means of a thief or a clean weighted, small-neck glass bottle. The bottle shall have a capacity of approximately 1 quart and shall be rinsed with the material being sampled. Lower the unstoppered bottle by means of a cord, chain, or rod to the bottom of the tank, and immediately withdraw to the surface. In order to obtain a representative cross-section sample of the material, the speed of lowering and raising the bottle shall be of uniform rate and regulated so that the bottle is just filled as it reaches the surface of the liquid.

F-3. *Inspection*.—

F-3a. *Army*.—Inspection shall be made in accordance with the requirements of U. S. Army Specification 50-0-1.

F-3b. *Navy*.—Inspection shall be made at the point of delivery unless otherwise specified in the contract or order.

F-4. *Tests*.—The laboratory tests shall be made in accordance with the following paragraphs. For Navy purchases, the tests shall be made at a Government laboratory unless otherwise specified in the contract or order.

F-4a. *Appearance*.—

F-4a(1). *Color*.—

F-4a(1)a. *Preparation of standard*.—Dissolve 1.245 gm. of potassium chloro-platinate (K_2PtCl_6), containing 0.500 gm. of platinum, and 1.000 gm. of crystallized cobaltous chloride ($CoCl_2 \cdot 6H_2O$) containing approximately 0.248 gm. of cobalt, in distilled water with 100 ml. of concentrated HCl. Dilute to 1 liter with pure distilled water. This solution is referred to as the 500 p.p.m. platinum standard. (See par. H-4.) Prepare a standard of 10 p.p.m. by diluting 1 ml of the 500 p.p.m. standard to 50 ml. with distilled water.

F-4a(1)b. *Procedure*.—Place both the 10 p.p.m. standard and the sample in 50-ml. tall form Nessler tubes to the same height. Look vertically downward through the liquid in the tubes upon a

white or mirrored surface which is placed at such an angle that light is reflected upward through the column of liquid. Compare the intensity of color of the sample and standard.

F-4a(2). *Suspended matter*.—Following inspection for color, note whether any suspended matter is observed when looking down the Nessler tube containing the sample.

F-4b. *Purity*.—Add 50 ml. of distilled water to a 250-ml. Erlenmeyer flask, add several drops of phenolphthalein indicator solution and titrate to a pink end point with 0.1N NaOH. Weigh accurately to plus or minus 0.0002 gm., approximately 5 gm. of the sample in a glass-stoppered weighing bottle. Remove the stopper, immerse bottle and stopper in the water and titrate with normal NaOH to the same end point. Calculate the purity as percent acetic acid as follows:

$$\text{Percent acetic acid} = \frac{6.005 \text{ VN}}{W}$$

where:

V = ml. of NaOH used in titrating sample

N = normality of NaOH used

W = grams of sample.

F-4c. *Freezing point*.—Transfer 15 ml. of sample to a test tube, 150 by 25 mm. approximate dimensions. Insert a 0° to 50°C. mercury thermometer graduated to 0.1°C., place the tube in an ice water bath and supercool to about 3°C. below the assumed freezing point. Remove the tube from the ice water and scratch the side of the tube with the thermometer and stir the contents. The supercooled liquid will partly crystallize and yield a mixture of liquid and solid. Dry the outside of the tube and stir constantly. Take thermometer readings to the nearest 0.05°C. The temperature rises quickly to a reading which is constant for approximately 30 seconds. This is taken as the freezing point. The thermometer shall be checked against one having a Bureau of Standards calibration.

F-4d. *Chlorides*.—Dissolve 2 ml. of the sample in 20 ml. of chloride-free distilled water, and add 1 ml. of concentrated HNO₃. Shake thoroughly and add a few drops of 1N silver nitrate solution. Note the appearance of any turbidity or opalescence which is indicative of the presence of chlorides.

F-4e. *Sulfates*.—Dissolve 2 ml. of the sample in 20 ml. of sulfate-free distilled water and add 1 ml. of concentrated HCl. Shake thoroughly and add a few drops of a 10 percent solution of barium chloride. Note the appearance of any turbidity or precipitate which is indicative of the presence of sulfates.

F-4f. *Formic and sulfurous acids*.—

F-4f(1). *Preparation of reagent*.—Dissolve 10 gm. of AgNO₃ in 400 ml. of distilled water. Add a mixture of 1 part of reagent grade ammonium hydroxide of sp. gr. 0.90 and 2 parts (by volume) of water, until the grayish brown precipitate which first forms is almost dissolved. Continue the addition of the ammonia solution dropwise with constant agitation until the brown haze just disappears. Caution: The presence of a minute excess of NH₄OH very greatly reduces the sensitivity of this reagent.

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F-4f(2). *Procedure.*—Place 2 ml. of the sample in a test tube and dilute with 18 ml. of pure distilled water. Exactly neutralize to litmus with the diluted NH_4OH used in preparing the ammoniacal silver nitrate. Add 10 ml. of ammoniacal silver nitrate and place the resulting mixture in a dark place for 20 minutes. Note the precipitation of silver or any darkening of color of the solution which is indicative of the presence of formic or sulfurous acids.

F-4g. *Heavy metals.*—

F-4g(1). Dissolve 10 ml. of the sample in 100 ml. of pure distilled water and add 5 ml. of concentrated HCl . Saturate the solution with H_2S gas. Note the appearance of a precipitate which is indicative of the presence of antimony, arsenic, bismuth, cadmium, copper, lead, mercury, or tin.

F-4g(2). Dissolve 10 ml. of the sample in pure distilled water and add 5 ml. excess NH_4OH using litmus paper indicator. Cool the solution and saturate with H_2S gas. Note the appearance of a precipitate which is indicative of the presence of cobalt, manganese, nickel, or zinc.

G. PACKAGING, PACKING, AND MARKING FOR SHIPMENT

G-1. *Packing.*—Unless otherwise specified, acetic acid shall be delivered in standard commercial containers, so constructed and protected as to conform to Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc., as applicable, and to insure acceptance by common or other carriers for safe transportation, at the lowest rate, to the point of delivery.

G-2. *Marking for shipment.*—In addition to any special marking required by the contract or order and marking to insure safe handling as required by Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc., shipments for the Army shall be marked in accordance with the requirements of U. S. Army Specification 100-2; for the Navy in accordance with the requirements of the Navy Shipment Marking Handbook.

H. NOTES

H-1. *Use.*—Acetic acid covered by this specification is intended for use in the manufacture of explosives.

H-2. Requests, requisitions, schedules, and contracts or orders should specify the title, number, and date of the specification.

H-3. This specification replaces Ordnance Department U. S. Army Tentative Specification AXS-793.

H-4. A 500 p.p.m. platinum color standard may be purchased as Harleco APHA Color Standards, Platinic Cobalt Chloride Series from the Hartman-Leddon Company, Philadelphia, Pa.

H-5. Information as to the availability of Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc., may be obtained from the Interstate Commerce Commission, Washington 25, D. C.

H-6. Copies of Joint Army-Navy specifications (required for Army purchases) and U. S. Army specifications may be obtained as indicated in the "Index of United States Army, Joint Army-Navy, and Federal Specifications Used by the War Department." Copies of this Index may be obtained from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Agencies within the War Department will obtain copies of Joint Army-Navy and U. S. Army specifications through established War Department channels. Both the title and identifying symbol number should be stipulated when requesting copies of specifications.

H-7. Copies of Joint Army-Navy specifications (required for Navy purchases), Navy Department specifications and the Navy Shipment Marking Handbook may be obtained upon application to the Bureau of Supplies and Accounts, Navy Department, Washington 25, D. C., except that Naval activities should make application to the Supply Officer in Command, Naval Supply Depot, Bayonne, N. J. Both the title and identifying symbol number should be stipulated when requesting copies of specifications.

H-8. Copies of this Joint Army-Navy specification (required for Army purchases) may be obtained from the Office, Chief of Ordnance, War Department, Washington 25, D. C.

Notice.—When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation, whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Army: O.
Navy: OS.