

# JAN-A-459

12 MARCH 1947

## JOINT ARMY-NAVY SPECIFICATION

### ACETIC ANHYDRIDE

(For Ordnance Use)

This specification was approved by the War Department and the Navy Department for use of procurement services of the Army and the 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.<sup>1</sup>

100-2—Standard Specification for Marking Shipments by Contractors.<sup>1</sup>

##### NAVY DEPARTMENT SPECIFICATION

General Specifications for Inspection of Material.<sup>2</sup>

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.<sup>2</sup>

##### 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 anhydride 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 parts per million of platinum. (See par. F-4a(1).)

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

E-2. *Purity.*—Minimum, 97.0 percent.

<sup>1</sup> Applicable only to Army purchases.

<sup>2</sup> Applicable only to Navy purchases.

**E-3. Chlorides.**—None. (See par. F-4c.)

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

**E-5. Heavy metals.**—When tested in accordance with the methods described in paragraph F-4e, 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 clean and dry 16-ounce glass stoppered bottle with the composite sample. If the material is shipped in tank cars, remove the sample (approx. 1 qt.) by means of a thief or a clean, dry and weighted, small-neck glass bottle. The bottle shall have a capacity of approximately one 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 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 chloroplatinate ( $K_2PtCl_6$ ), 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 one liter with pure distilled water. This solution is referred to as the 500 ppm platinum standard. (See par H-4.) Prepare a standard of 10 ppm by diluting 1 ml of the 500 ppm standard to 50 ml with distilled water.

**F-4a(1)b. Procedure.**—Place both the 10 ppm 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.**—To a 500-ml dry glass-stoppered Erlenmeyer flask, add exactly 50 ml of approximately 0.5 N carbonate-free NaOH and stopper the flask. To a 250-ml glass-stoppered Erlenmeyer flask, add 20 ml of dry freshly distilled aniline, and stopper the flask. By means of a Lunge weighing bottle, accurately weigh to  $\pm 0.0002$  gm, 0.9 to 1.0 gm of sample into the 500-ml flask containing NaOH and 1.6 to 1.7 gm into the 250-ml flask containing aniline. The samples should be added dropwise and the flasks swirled to prevent local heating. Stopper each flask immediately after sample is added. Allow to stand for 3 to 5 minutes and wash down the flask containing NaOH with 100 ml of neutral (to phenolphthalein) distilled water. Titrate with N/2 HCl to a phenolphthalein end point. Allow the flask containing the aniline, to cool, and dilute the contents with 100 ml of neutral (to phenolphthalein) solution of 75 ml of methyl alcohol and 25 ml of water. Titrate with N/2 carbonate-free NaOH to a phenolphthalein end point. Titrate a blank solution consisting of 100 ml of the alcohol solution and 20 ml of the aniline using 0.1N carbonate-free NaOH. Calculate the percent purity as acetic anhydride as follows:

$$\text{Percent acetic anhydride} = 10.21 \left\{ \frac{V_1 N_1 - V_2 N_2}{W_1} - \frac{V_3 N_1 - V_4 N_1}{W_2} \right\}$$

where

$V_1$  = ml NaOH added to first flask = 50.

$V_2$  = ml HCl used in back titration of contents of first flask.

$V_3$  = ml NaOH used in titrating second flask.

$V_4$  = ml NaOH used in blank.

$N_1$  = normality of NaOH add to first flask and used in titration of contents of second flask.

$N_2$  = normality of HCl used in back titration.

$N_3$  = normality of NaOH used in blank.

$W_1$  = gm of sample used in first flask (with NaOH).

$W_2$  = gm of sample used in second flask (with aniline).

**F-4c. Chlorides.**—Dissolve 2 ml of the sample in 20 ml of chloride free distilled water, and add 1 ml of concentrated HNO<sub>3</sub>. 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-4d. 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-4e. Heavy Metals.**—

**F-4e(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<sub>2</sub>S gas. Note the appearance of a precipitate

which is indicative of the presence of antimony, arsenic, bismuth, cadmium, copper, lead, mercury or tin.

F-4e(2). Dissolve 10 ml of the sample in pure distilled water and add 5 ml. excess  $\text{NH}_4\text{OH}$  using litmus paper indicator. Cool the solution and saturate with  $\text{H}_2\text{S}$  gas. Note the appearance of a precipitate which is indicative of the presence of cobalt, manganese, nickel, and zinc.

#### G. PACKAGING, PACKING, AND MARKING FOR SHIPMENT

G-1. *Packing*.—Unless otherwise specified, acetic anhydride shall be packed in standard commercial containers, so constructed and protected to conform to applicable regulations of the Interstate Commerce Commission, Regulations for the Transportation of Explosives and Other Dangerous Articles, etc., 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 Marking Handbook.

#### H. NOTES

H-1. *Use*.—Acetic anhydride 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-794.

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

H-5. 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-6. 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,