

JAN-D-664

27 AUGUST 1948

NATIONAL MILITARY ESTABLISHMENT SPECIFICATION DINITROMETHYLANILINE IN SULFURIC ACID

This specification was approved by the Departments of the Army, the Navy, and the Air Force for use of procurement services of the respective Departments.

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:

JOINT ARMY-NAVY SPECIFICATION

JAN-A-179—Acid, Sulfuric, and Oleum.

U. S. ARMY SPECIFICATIONS

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

50-11-107—Dinitromethylaniline.

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 a solution of dinitromethylaniline in sulfuric acid.

C. MATERIAL AND WORKMANSHIP

C-1. The solution shall be manufactured by dissolving dinitromethylaniline conforming to U. S. Army Specification 50-11-107, in sulfuric acid conforming to Grade I of Joint Army-Navy Specification JAN-A-179. The solution shall be olive green in color.

D. GENERAL REQUIREMENTS

D-1. See section E.

E. DETAIL REQUIREMENTS

E-1. *Dinitromethylaniline content.*— 20.0 ± 1.5 percent.

¹ Applicable only to Army purchases.

² Applicable only to Navy purchases.

E-2. *Sulfuric acid concentration*.— 91.5 ± 1.5 percent.

E-3. *Inorganic chlorides*.—Maximum, 0.015 percent.

F. METHODS OF SAMPLING, INSPECTION, AND TESTS

F-1. *Size of lot*.—Maximum, 120,000 pounds.

F-2. *Sampling*.—Take a sample of approximately 1 pint from each tank car by means of a thief and transfer it to a glass-stoppered bottle as rapidly as possible to prevent absorption of moisture. If preferred, remove a portion from each car by lowering an empty narrow-neck, glass-stoppered bottle (stopper removed) at a uniform rate so that it will be filled on arriving at the bottom of the car. Stopper and seal the bottle, and label this to show the name of the material, tank car number, manufacturer, plant, contract or order number, and number of pounds in the lot. All acceptance tests shall be made on the sample representative of the lot.

F-3. *Inspection*.—Inspection shall be made in accordance with U. S. Army Specification 50-0-1 and shall be made at the place of manufacture, unless otherwise specified in the contract or order.

F-4. *Tests*.—

F-4a. *Dinitromethylaniline content*.—By means of a Lunge acid-weighing pipet transfer a weighed portion of approximately 0.8 gm. of the sample to a 250-ml. beaker. Place the beaker in an ice bath. Add 30 ml. of distilled water at a temperature of 0° to 3°C . to the beaker, mix well, and crush the precipitate with a glass rod having a flattened end. Transfer the precipitate to a tared Gooch crucible, using not more than 60 ml. of distilled water at 0° to 3°C . to effect the transfer and for subsequent washing of the precipitate. Retain the filtrate and washings for the determination of the concentration of sulfuric acid as directed under paragraph F-4b. Dry the crucible and contents to constant weight in an oven maintained at 108° to 112°C ., cool in a desiccator and weigh. Calculate the increase in weight as percent dinitromethylaniline.

F-4b. *Sulfuric acid concentration*.—Transfer the combined filtrate and washings from the determination of dinitromethylaniline (see par. F-4a) to a beaker and titrate with approximately N/3 sodium hydroxide solution, using phenolphthalein as the indicator. Calculate the percent concentration of the sulfuric acid as follows:

$$\text{Percent sulfuric acid concentration} = \frac{490.4VN}{(100-D)W}$$

where: V = ml. of sodium hydroxide solution used
 N = normality of sodium hydroxide solution
 D = percent dinitromethylaniline (see par. F-4a)
 W = gm. of sample.

F-4c. *Inorganic chloride*.—Weigh a portion of approximately 50 gm. of the sample in a glass-stoppered weighing bottle and transfer to a 2-liter beaker containing 1200 ml. of distilled water. Wash out the weighing bottle thoroughly with distilled water, adding the washings to the solution, and bring the mixture to a volume of 1500 ml. with distilled water. Filter the mixture on a Buchner

funnel. Wash the precipitate on the funnel with several 25-ml. portions of distilled water at a temperature of 20° to 25°C., adding the washings to the filtrate. Add 5 ml. of concentrated nitric acid, and slowly, with constant stirring, 5 ml. of a 5-percent silver nitrate solution. Heat the mixture to approximately 60°C., allow the precipitate to settle, and add a few more drops of silver nitrate solution. Repeat the operation if additional precipitate forms. When precipitation is complete, cover the beaker, and set aside in a dark place for a few hours or preferably overnight. Decant the clear solution through a tared Gooch crucible, and wash the precipitate in the beaker by decantation with a solution containing 0.05 gm. of silver nitrate per liter. Transfer the precipitate to the crucible and wash with 1-percent nitric acid solution. Treat the precipitate with a few drops of concentrated nitric acid until any gummy material disappears. Dry the crucible and contents for 15 minutes in an oven maintained at 100° to 105°C. and then for 1 hour at 130° to 150°C., cool in a desiccator and weigh. Calculate the increase in weight to percent sodium chloride as follows:

$$\text{Percent sodium chloride} = \frac{40.78A}{W}$$

where: A = gm. of precipitate
W = gm. of sample.

G. PACKAGING, PACKING, AND MARKING FOR SHIPMENT

G-1. The solution shall be shipped in tank cars.

G-2. *Marking.*—Shipments shall be marked to insure safe handling as required by Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc., and in addition 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. Any special marking shall be as specified in the contract or order.

H. NOTES

H-1. *Use.*—Dinitromethylaniline solution in sulfuric acid covered by this specification is intended for use in the manufacture of tetryl.

H-2. This specification replaces Picatinny Arsenal Tentative Specification PXS-978.

H-3. Information as to the availability of Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc., may be obtained upon application to the Interstate Commerce Commission, Washington 25, D. C.

H-4. Copies of National Military Establishment and Joint Army-Navy specifications (required for Army and Air Force purchases) and U. S. Army specifications may be obtained as indicated in the "Index of United States Army, Joint Army-Navy, and Federal Specifications and Standards." Copies of this Index may be obtained from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Agencies within the Department of the Army, and the Department of the Air Force will

[JAN-D-664]

obtain copies of National Military Establishment, Joint Army-Navy, and U. S. Army specifications through established departmental channels. Both the title and identifying symbol number should be stipulated when requesting copies.

H-5. Copies of National Military Establishment and 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 Center, Norfolk 11, Va. Both the title and identifying symbol number should be stipulated when requesting copies.

H-6. Copies of this specification (required for Army purchases) may be obtained from the Office, Chief of Ordnance, Department of the Army, 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.

[JAN-D-664]