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SUPERSEDING
Picatinny Arsenal
Tentative Specifications
PXS–1271
9 Feb. 1948

NATIONAL MILITARY ESTABLISHMENT SPECIFICATION

DIMETHYLPHTHALATE
(For Use in Explosives)

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.

1. SCOPE AND CLASSIFICATION

1.1 This specification covers dimethylphthalate for use in the manufacture of propellent powder.

2. APPLICABLE SPECIFICATIONS AND OTHER PUBLICATIONS

2.1 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 SPECIFICATIONS

General Specifications for Inspection of Material [2]

(*Army.*—Copies of specifications should be obtained from the procuring agency or as directed by that agency. Both the title and identifying number or symbol should be stipulated when requesting copies.)

(*Navy.*—Copies of National Military Establishment specifications and Navy Department specifications may be obtained upon application to the Bureau of Supplies and Accounts, Navy Department, Washington 25, D. C., except that activities of the Armed Forces should make application to the Supply Officer in Command, Naval Supply Center, Norfolk 11, Va. Both the title and identifying number or symbol should be stipulated when requesting copies.)

[1] Applicable only to Army purchases.
[2] Applicable only to Navy purchases.

2.2 Other publication.—The following publication, of the issue in effect on date of invitation for bids, forms a part of this specification:

BUREAU OF SUPPLIES AND ACCOUNTS PUBLICATION

Navy Shipment Marking Handbook [2]

(Copies of the Navy Shipment Marking Handbook may be obtained upon application to the Bureau of Supplies and Accounts, Navy Department, Washington 25, D. C.)

3. REQUIREMENTS

3.1 Color.—No darker than the standard (See 4.41).

3.2 Specific gravity at 15.5°/15.5° C.—1.96±0.010.

3.3 Ester content (as dimethylphthalate).—Minimum, 99 percent.

3.4 Acidity (as phthalic acid).—Maximum, 0.03 percent.

3.5 Ash.—Maximum 0.01 percent.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 LOT.—Maximum, 50,000 pounds.

4.2 Sampling.—Ten percent, but in no case more than 10 or less than 3, of the containers shall be selected by the inspector in such manner as to be representative of the lot. If there are fewer than three containers in the lot, all the containers shall be sampled. Mix the material thoroughly to a uniform consistency throughout and remove from each selected container sufficient material to form a primary sample of approximately 8 ounces. Place the sample in an

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airtight container and label each primary sample so that the container from which it was taken can be easily identified. From each primary sample remove a sufficient quantity of material to form a composite sample of about 8 ounces. Mix the sample thoroughly and place in an airtight container. Label the container to show the name of the material, manufacturer, plant, contract or order number, lot number, and lot size. All acceptance tests shall be made on the composite sample. However, if it becomes apparent during sampling that the lot is not uniform, the inspector may require that any primary sample be tested for compliance with the requirements of the specification. All primary samples shall be held for possible future examination should the composite sample fail to meet the requirements of the specification.

4.3 **Inspection.**—Inspection shall be made in accordance with U. S. Army Specification 50-0-1 and shall be made at the point of delivery unless otherwise specified.

4.4 **Tests.**—Tests shall be made as follows:

4.4.1 *Color.*—Add 0.5 ml. of 0.1 N iodine solution to 100 ml. of distilled water. Compare the color with 25 ml. of the sample.

4.4.2 *Specific gravity.*—Determine the specific gravity at 15.5°/15.5° C. by means of a pycnometer or Westphal balance.

4.4.3 *Ester content (as dimethylphthalate).*—Transfer 1.5 gm. of the sample to a 250 ml. pyrex or similar flask and add 50 ml. of 0.5 N alcoholic KOH by means of a calibrated buret or pipet (0.5 N alcoholic KOH is prepared by adding 28 gm. of reagent-grade KOH to 1 liter of 95-percent alcohol). Fit the flask to a reflux condenser by means of ground joints and heat on a water bath until the precipitation appears to be complete. Add approximately 15 ml. of distilled water to dissolve the potassium phthalate and reflux on a water bath for not less than 45 minutes with occasional whirling of the flask. Wash down the sides of the reflux condenser and the ground joints with approximately 25 ml. of the distilled water. Cool the solution to 25° C. and titrate with 0.5 N HCl using phenolphthalein indicator. (See 6.2.) At the same time, run a blank determination on 50 ml. of alcoholic KOH which has been carried through the complete process. Calculate the ester content as percent dimethylphthalate as follows:

$$\text{Percent dimethylphthalate} = \frac{9.709(V-v)N}{W}$$

where

N = normality of the acid used.
V = ml. of acid used to titrate blank.
v = ml. of acid used to titrate excess of KOH after saponification.
W = gm. of sample.

4.4.4 *Acidity.*—Measure 100 ml. of ethyl alcohol into a flask, add 3 drops of phenolphthalein indicator, and titrate the alcohol to a faint pink color with 0.1 N NaOH. Add an accurately measured 50 ml. portion of the sample to the alcoholic solution. Titrate the mixture with 0.1 N NaOH. Calculate the percent acidity as phthalic acid as follows:

$$\text{Percent phthalic acid} = \frac{0.166\,VN}{G}$$

where

V = ml. NaOH required for titration of the sample.
N = normality of NaOH solution.
G = specific gravity of sample.

4.4.5 *Ash.*—Weigh a portion of approximately 10 gm. of the sample in an accurately tared porcelain crucible. Evaporate nearly to dryness over a low flame or on a hot plate. Ignite the residue to constant weight at a red heat. Cool the crucible in a desiccator and weigh. Calculate the increase in weight as percent ash.

4.5 **Resubmission and retests.**—If the composite sample or any primary sample subject to test fails to pass the tests, the lot shall be rejected. The contractor shall have the option of having a partial or complete analysis made on each container in the lot at no expense to the Government. The contractor may then remove the defective portion of the lot, and resubmit the lot for acceptance provided complete replacement of the defective portion can be made to the satisfaction of the inspector. For acceptance of the resubmitted lot, new samples shall be selected as specified in 4.2 and shall pass the test requirements specified herein.

5. PREPARATION FOR DELIVERY

5.1 **Packing.**—Unless otherwise specified, dimethylphthalate shall be delivered in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation, at the lowest rate, to the point of delivery.

5.2 **Marking.**—In addition to any special marking required by the contract or order, 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.

6. NOTES

6.1 **Ordering data.**—Purchase instruments should specify the title, the number, and the date of the specification.

6.2 **Phenolphthalein indicator.**—Phenolphthalein indicator may be prepared by dissolving one gm. of phenolphthalein in 100 ml. of 85-percent (by volume) ethyl alcohol.

Custodian:
Army—Ordnance Department.
Other interest:
Navy—OS.