

MIL-D-22346B (OS)
19 March 1973

SUPERSEDING

MIL-D-22346A (Wep)
21 December 1961

MILITARY SPECIFICATION

DI-NORMAL-PROPYL ADIPATE

*This specification has been approved by the Naval Ordnance Systems Command,
Department of the Navy.*

1. SCOPE

1.1 This specification covers di-normal-propyl adipate for use in propellants.

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 the specification to the extent specified herein.

STANDARDS

Military

MIL-STD-129

Marking for Shipment and Storage

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials

ASTM E 200

Preparation, Standardization, and Storage of
Standard Solutions for Chemical Analysis

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(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa. 19103.)

3. REQUIREMENTS

3.1 Acidity. The acidity shall be 0.1 percent maximum when determined in accordance with 4.4.1.

3.2 Ester content. The ester content shall be 99.0 percent minimum when determined in accordance with 4.4.2 and corrected for acidity as determined in 4.4.1.

3.3 Specific gravity. The specific gravity shall be not greater than 0.982 nor less than 0.972 at 20°/20° centigrade (C) when determined in accordance with 4.4.3.

3.4 Freezing point. The freezing point shall be not lower than -19° C nor higher than -13° C when determined in accordance with 4.4.4.

3.5 Refractive index. The refractive index shall be not greater than 1.4298 nor less than 1.4290 when determined in accordance with 4.4.5.

3.6 Color. The color shall be APHA 30 maximum or natural water white when determined in accordance with 4.4.6.

3.7 Ash. The ash shall be 0.002 percent maximum when determined in accordance with 4.4.7.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier 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 the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Sampling.

4.2.1 Lot. A lot shall consist of di-normal-propyl adipate manufactured in one batch by the same processing, unit, chemical, or physical mixing process, which is intended to make the final product uniform.

4.2.2 Samples. At least 10 percent of the containers shall be sampled and a composite prepared therefrom for analysis.

4.3 Acceptance criteria. Failure of any sample to meet any requirement of this specification shall be cause for rejection of the lot.

4.4 Test methods.

4.4.1 Acidity. Weigh accurately to 0.1 milligram approximately 25 grams of the sample into a 125-milliliter (ml) Erlenmyer flask and dissolve in 50 ml of ethyl alcohol. Add 3 drops of phenolphthalein and titrate to a persistent faint pink color with 0.1N sodium hydroxide solution prepared in accordance with ASTM E-200, paragraphs 13 through 18. Make a blank titration on 50 ml of the ethyl alcohol used to dissolve the sample. Calculate the percent acidity as adipic acid according to the following:

$$\text{Percent adipic acid} = \frac{7.3073 (A - B)N}{W}$$

where

A = ml of NaOH solution required for titration of sample

B = ml of NaOH solution required for titration of blank

N = normality of NaOH solution

W = weight of sample in grams.

4.4.2 Ester content as di-normal-propyl adipate. Transfer 1.5 grams of sample to a 250-ml Pyrex erlenmeyer or similar flask fitted with a ground glass joint. Add 50 ml of 0.5N alcoholic potassium hydroxide (KOH) by means of a calibrated buret or pipet. (0.5N alcoholic KOH is prepared by adding 28 grams of reagent grade KOH to 1 liter of 95 percent alcohol.) Fit the flask to a reflux condenser fitted with an NaOH-filled drying tube. Heat on water bath until the precipitation appears to be complete. Add approximately 15 ml of distilled water to dissolve the potassium adipate and reflux on a water bath for not less than 45 minutes with occasional swirling of the flask. Wash down the sides of the reflux

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condenser and the ground joints with approximately 25 ml of distilled water. Cool the solution to 25° C and titrate with 0.5N hydrochloric acid using phenolphthalein indicator. 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 di-normal-propyl adipate as follows:

$$\text{Percent di-normal-propyl adipate} = \left(\frac{11.515(V - n)N}{W} \right) - (A \times 1.58)$$

where

- N = normality of the acid used
- V = ml of acid used to titrate blank
- n = ml of acid used to titrate excess of KOH
- W = weight of sample in grams
- A = percent acidity as adipic acid.

4.4.3 Specific gravity. Determine the specific gravity at 20°/20° C by means of a pycnometer or Westphal balance.

4.4.4 Freezing point. Heat a test tube moderately over a flame to remove moisture. Place about 35 grams of sample in test tube and place inner tube in outer tube of apparatus (figure 1). Place assembly in dry ice-acetone bath, and stir sample with a reciprocating action until first crystal forms. This is the freezing point. Remove tube from bath and, continuing the reciprocating stirring, note disappearance of crystal.

4.4.5 Refractive index. Determine the refractive index at 25° C by use of an Abbe or dipping refractometer.

4.4.6 Color.

4.4.6.1 Standard preparation. Dissolve 1.245 grams of potassium chloroplatinate (K_2PtCl_6) and 1 gram of crystallized cobaltous chloride ($CoCl_2 \cdot 6H_2O$) in distilled water containing 100 ml of 37 percent hydrochloric acid, and make up to 1000 ml in a volumetric flask with distilled water. This is best done in two steps for colors below 50 in order to avoid measurement of extremely small volumes of the color standard. See table I for dilution ratios. Store the color standards in glass-stoppered bottles, preferably Pyrex.

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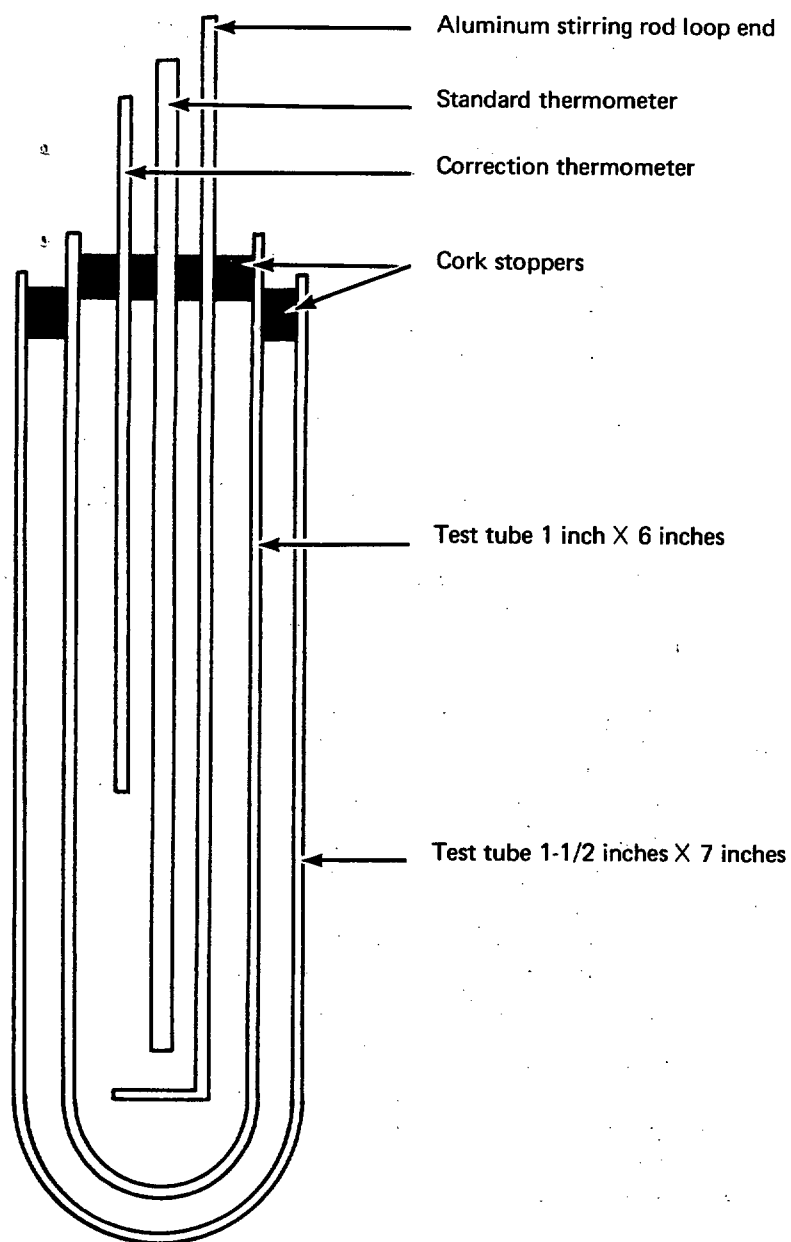


FIGURE 1. FREEZING POINT APPARATUS

Table I

HAZEN OR APHA COLOR STANDARDS

| Color standards | Milliliters of 500 standard | Milliliters of distilled water |
|-----------------|-----------------------------|--------------------------------|
| 500 | 100 | 0 |
| 450 | 90 | 10 |
| 400 | 80 | 20 |
| 350 | 70 | 30 |
| 300 | 60 | 40 |
| 250 | 50 | 50 |
| 200 | 40 | 60 |
| 150 | 30 | 70 |
| 100 | 20 | 80 |
| 50 | 10 | 90 |
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| 50 | 100 | 0 |
| 40 | 80 | 20 |
| 30 | 60 | 40 |
| 20 | 40 | 60 |
| 10 | 20 | 80 |
| 0 | 0 | 100 |

4.4.6.2 Comparison. Compare the color of the sample visually with an equal amount of the Hazen color standards. This may conveniently be done by placing the Hazen standards in Nessler tubes or in a uniformly shaped bottle and by placing the sample in a spare bottle of the same type glass and shape. It is suggested that 125-ml square glass-stoppered bottles be used although any style or shape bottle or Nessler tube may be used for comparison. Report as the color, the number of the standard that most nearly matches the sample. In the event that the color lies midway between two standards, report the darker of the two.

4.4.7 Ash. Weigh a portion of approximately 10 grams 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 as follows:

$$\text{Percent ash} = \frac{\text{Increase in weight} \times 100}{\text{Weight of sample}}$$

5. PREPARATION FOR DELIVERY

5.1 Packing. Packing shall be level C. Unless otherwise specified, di-normal-propyl adipate 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. Unless otherwise specified, containers shall be marked in accordance with MIL-STD-129.

6. NOTES

6.1 Ordering data. Procurement documents should specify the following:

- a. Title, number, and date of this specification
- b. Quantity required.

Custodian:
Navy - OS

Preparing activity:
Navy - OS
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