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AUTHORITY

USNWC ltr, 30 Aug 1974

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SPECIFICATION

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1. COMPONENT/PART NAME PER GENERIC CODE: Propulsion Parts & Materials, Solid Fuel Engines, Propellants	2. PROGRAM OR WEAPON SYSTEM: Multiple
3. DATE OF: ISSUE	DAY 13 MO 10 YR 67
4. ORIGINATOR'S SPECIFICATION TITLE: Purchase Description - N-Phenylmorpholine	5. ORIGINATOR'S SPEC. NO.: WS 7657
	3. DATE OF: REVISION
	6. SPECIFICATION IS: ☐ DRAFT ☐ PRELIMINARY ☒ FINAL

7. THIS SPECIFICATION COMPLEMENTS REPORT NO:

8. TYPE OF SPECIFICATION

- ☒ (A) GENERAL PRODUCT REQUIREMENTS FOR A FAMILY OF PARTS - PROCUREMENT DOCUMENT
- ☐ (B) INDIVIDUAL DETAIL PARTS DOCUMENT; STDS BOOK PAGES - FOR PROCUREMENT
- ☐ (C) DETAIL INSPECTION, PROCESS CONTROL, AND/OR TEST PROCEDURES FOR SPECIFIC PARTS
- ☐ (D) PROCESS (PAINTING, WELDING, FINISHING, HEAT TREATING ETC.) APPLICABLE TO MANY PARTS
- ☐ (E) SPEC. FOR PERFORMANCE, RELIABILITY, AND/OR ENVIRONMENT FOR ASSEMBLIES, EQUIPMENTS, SUBSYSTEMS AND SYSTEMS
- ☐ (F) PERFORMANCE AND APPLICATION DATA FOR DESIGN ENG USE ON PARTS - NOT FOR PROCUREMENT
- ☐ (G) OTHER (DETAIL IN 10.)

NOTE: WHEN MULTIPLE FUNCTIONS ARE COMBINED IN ONE DOCUMENT, CHECK ALL APPLICABLE SQUARES.

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1961 Book of ASTM Std. Part 27			x	

DDC

JAN 27 1969

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13. SPEC. NO. 565.70.00.00-X7-12

11. SIGNED R. S. Harper 11/16/68

12. CONTRACTOR NWC/CL | SUBCONTRACTOR

NOTICES PAGE

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Code Ident 30003 WS 7657

NAVAL AIR SYSTEMS COMMAND

DEPARTMENT OF THE NAVY

PURCHASE DESCRIPTION

N-PHENYLMORPHOLINE

1. SCOPE

1.1 Scope. This purchase description covers one grade of N-phenylmorpholine.

2. APPLICABLE DOCUMENTS.

2.1 The following document of the issue in effect on date of invitation for bids or request for proposal forms a part of this document 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.)

FSC 6810

2.2 Other publications. The following document forms a part of this document 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)

1961 Book of ASTM Standards; Part 27	Test-Method ASTM D1303-55, "Total Chlorine in Vinyl Chloride Polymers and Copolymers".

(ASTM Publications are published by the American Society for Testing and Materials, Philadelphia 3, Pennsylvania.)

3. REQUIREMENTS.

3.1 Preproduction sample. Unless otherwise specified (see 6.2), a preproduction sample shall meet all requirements of this document. The preproduction sample shall be prepared using the same methods and procedures proposed for production. Any production prior to acceptance of the preproduction sample shall be at the risk of the supplier.

3.2 Data. No data is required by this document or by referenced documents in section 2 unless specified in the contract or purchase order.

3.3 Compliance to documents. N-phenylmorpholine shall conform to the requirements herein and to the applicable requirements of documents listed in section 2.

3.4 Product characteristics and performance. When tested in accordance with 4.7 of this document, N-phenylmorpholine shall meet the following product characteristics and performance.

3.4.1 Chemical analysis. The chemical analysis of the material shall be specified in Table I.

Table I Chemical Analysis

Characteristics	Minimum	Maximum
N-phenylmorpholine, total alkalinity, %	99.0	---
N-phenylmorpholine, tertiary amine, %	98.0	---
Chlorine, %	---	0.10
Melting point, °C.	50	54
Moisture, %	---	0.10

3.5 Workmanship. The N-phenylmorpholine shall be uniform in quality, free from foreign materials, and shall be manufactured under conditions and procedures standard in the industry.

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 the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in this document where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Lot. A lot shall consist of material produced at one plant with no change in formulation or process. If manufacture is by batch process, each batch shall constitute a lot. A batch shall be as defined in 6.3.

4.3 Acceptance sampling. The number of containers to be chosen at random for acceptance sampling shall be equal to the square root of the total number of containers in the lot. If the number thus obtained is not a whole number, the number of containers to be sampled shall be increased to the next

higher whole number. In no case, however, shall the number of containers to be sampled be less than seven (unless there are less than seven containers in the lot, in which case, each container shall be sampled).

4.3.1 Primary sample. From each selected container, a sample shall be taken from three or more places throughout the container. The total weight of the samples taken from each container shall weigh at least 50 grams (gm). Each sample thus taken shall be mixed thoroughly, placed in a clean dry container, and labeled to identify the material name, original container designation, contract number, and lot number.

4.3.2 Composite sample. Each primary sample shall be subdivided to prepare a composite sample (not in excess of 100 gm). Primary material not used shall be returned to the primary sample container. After mixing the composite sample thoroughly, the composite sample shall be placed in a clean, dry container and sealed. The composite sample shall be identified with the material name, container designation, contract number, and lot number. All specified chemical tests shall be made on this composite sample representing the lot. Failure of the composite sample to pass all of the tests herein shall result in rejection of the lot represented.

4.4 Classification of tests. Inspection and testing of N-phenylmorpholine shall be classified as follows:

(a) Preproduction tests.

(b) Quality conformance tests.

4.5 Preproduction tests. Preproduction tests shall be conducted only on the preproduction sample and shall consist of all examinations and tests specified in 4.6.

4.6 Quality conformance tests. Quality conformance tests for acceptance of the N-phenylmorpholine shall consist of the following tests:

Characteristics	Test
N-phenylmorpholine, total alkalinity	4.7.1
N-phenylmorpholine, tertiary amine	4.7.2
Chlorine	4.7.3
Melting point	4.7.4
Moisture	4.7.5

4.7 Tests. The following procedures shall be used to determine that the requirements of this document have been met. Any proposed change in test procedures or equipment shall necessitate, before adoption, prior approval of the procuring activity. In case of dispute between the results from any proposed method or equipment and what is cited herein, the results using the methods and the equipment specified in this document shall prevail. Unless otherwise specified, all tests shall be run in duplicate. The average of the two results shall be taken as the test result.

4.7.1 Percent N-phenylmorpholine by total alkalinity.

4.7.1.1 Procedures.

WARNING

This procedure involves the use of perchloric acid. Personnel using this reagent must be made aware of its potential hazards.

(a) Transfer approximately 5 gm of the molten sample to a tared 100-milliliter (ml), glass-stoppered, volumetric flask. Reweigh the flask to the nearest 0.0001 gm. The difference in weight is the weight of the sample.

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(b) Add 50 ml of redistilled acetonitrile to the flask and swirl to effect complete solution. Dilute to the mark with acetonitrile and mix thoroughly.

(c) Into each of two 500-ml, glass-stoppered Erlenmeyer flasks, pipet 10 ml of the sample dilution.

(d) Pipet 10 ml of redistilled acetonitrile into each of two additional 500-ml Erlenmeyer flasks and reserve for the blank determination.

(e) To each of the flasks add 100 ml of glacial acetic acid which has been neutralized to the first green color using 0.1N (Normal) perchloric acid ($HClO_4$) and 1.0 percent crystal violet indicator in acetic acid.

(f) Add one or two drops of crystal violet indicator to each flask and titrate with standard 0.1N $HClO_4$ to the first green end point.

$$\text{Percent N-phenylmorpholine} = \frac{(A-B)\ N \times 16.32}{C \times 0.1}$$

Where: A = Volume of N $HClO_4$ required for sample, ml.
B = Volume of N $HClO_4$ required for blank, ml.
C = Weight of sample taken, gm.
N = Normality of $HClO_4$.

4.7.1.2 Acceptance criteria. For the lot represented to pass the N-phenylmorpholine test, the value obtained for the percent N-phenylmorpholine by total alkalinity shall be not less than the value specified in 4.6.1.

4.7.2 Percent N-phenylmorpholine by tertiary amine.

4.7.2.1 Procedure.

(a) Into each of two 500-ml, glass-stoppered, Erlenmeyer flasks, pipet 10 ml of the sample dilution (4.7.1.1 (b)).

(b) Into each of two additional 500-ml Erlenmeyer flasks, pipet 10 ml of redistilled acetonitrile and reserve for the blank determination.

(c) While swirling slowly, add 20 ml of refined acetic anhydride to each of the blank and sample flasks and allow to stand at room temperature for 5 to 10 minutes.

(d) Into each of the flasks, introduce 100 ml of glacial acetic acid which has been neutralized with 0.1N $HClO_4$ to the first green end point of crystal violet indicator.

(e) Swirl the contents of the flask, add one or two drops of crystal violet indicator and titrate with standard 0.1N $HClO_4$ to the first green end point.

$$\text{Percent N-phenylmorpholine} = \frac{(A-B)\ N \times 16.32}{C \times 0.1}$$

Where: A = Volume of N $HClO_4$ required for the sample, ml.
B = Volume of N $HClO_4$ required for the blank, ml.
C = Weight of sample taken, gm.
N = Normality of $HClO_4$.

4.7.2.2 Acceptance criteria. For the lot represented to pass the N-phenylmorpholine test, the value obtained for the percent N-phenylmorpholine by tertiary amine shall be not less than the value specified in 4.3.1.

4.7.3 Chlorine. The percentage of chlorine specified in 3.4.1 shall be determined in accordance with the procedure given in Test Method D1303-55 "Total Chlorine in Vinyl Chloride Polymers and Copolymers" (Part 27 of ASTM, page 472), except use ferric alum indicator in place of ferric nitrate indicator, and electrical ignition of the Parr bomb in place of ignition by use of a Bunsen burner.

4.7.3.1 Acceptance criteria. For the lot represented to pass the chlorine test, the value obtained for the percent chlorine shall be no greater than the value specified in 4.3.1.

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4.7.4 Melting point.

4.7.4.1 Procedure. Place a few crystals of N-phenylmorpholine on an 18-millimeter (mm) diameter microcover glass. Cover the crystals with an identical cover glass and transfer to the cleaned heating stage of a Fisher-Johns melting point apparatus or approved equivalent (4.7.4.2). Turn on the apparatus; bring the temperature to 45 degrees Centigrade (°C) (113 degrees Fahrenheit (°F)), and hold the apparatus at that temperature for at least 5 minutes. Then adjust the reading of the current-input dial to give a temperature rise rate of 0.8 to 1.0°C (1.4 to 1.8°F) per minute as indicated by the attached thermometer. With the aid of the illuminating and magnifying unit, observe the melting point at which the last crystal of N-phenylmorpholine liquefies.

4.7.4.2 Apparatus. Melting Point Apparatus, Fisher-Johns, available from the Fisher Scientific Company, Pittsburgh, Pennsylvania.

4.7.4.3 Acceptance criteria. For the lot represented to pass the melting point test, the value obtained for the temperature of the melting point shall be within the range in 4.3.1.

4.7.5 Moisture.

4.7.5.1 Apparatus. The apparatus used for determination of moisture content of the sample shall be an Aquameter, Model KF-2 or KF-3, Beckman Instruments, Inc., Fullerton, California or an approved equivalent. The Aquameter shall be prepared for operation as described in the technical manual furnished by the manufacturer (Beckman Instruments, Inc.). Use of an alternate equivalent item of equipment approved by the procuring activity will necessitate use of the specific technical manual prepared by the manufacturer.

4.7.5.2 Reagents.

(a) Karl Fischer reagent. Karl Fischer reagent must have a strength such that each milliliter of Karl Fischer reagent corresponds to 0.0014-0.0023 gm of water. Dilute 750 ml of commercially available stabilized Karl Fischer reagent (with water equivalent of 0.005-0.007 gm/ml) to 2000 ml with absolute methanol (0.1 percent water, maximum). Mix well and allow to stand overnight before use. Determine the water equivalent (A) of this solution as follows:

1. Use sodium tartrate dihydrate ($Na_2C_4H_4O_6 \cdot 2H_2O$) as a primary standard (with a water content of 15.66 percent) for standardizing Karl Fischer reagent. If the water content value is in question, it may be determined by heating some of the salt at 150°C (302°F) for 3 hours. Should the value (as determined) differ from the theoretical value of 15.66, then the experimental value shall be used in the determination of water equivalent (A) of the Karl Fischer reagent; i.e., instead of the 15.66 in the formula below, the factor should be 10P where P is percentage moisture (as determined). Rapidly transfer 0.090-0.110 gm (weighed to the nearest 0.0001 gm) of reagent-grade $Na_2C_4H_4O_6 \cdot 2H_2O$ to the titration vessel.

2. Titrate to an end point in the same manner as with the sample. (See 4.7.5.3.)

3. Repeat the standardization procedure until three successive results agree within five parts per thousand.

4. If the indicated water equivalent (A) of the Karl Fischer reagent is less than 0.0014 gm of water per ml of Karl Fischer reagent, it may be due to the presence of too much water in the absolute methanol used. In this case, distill the methanol from metallic calcium or calcium hydride. Passing the methanol

through a column of Molecular Sieves, Type 4A, or equivalent may also reduce the water content of the methanol sufficiently. (Molecular Sieves are a product of the Linde Company, a division of Union Carbide Corporation, New York City, New York.)

$$\text{Water equivalent (A)} = \frac{0.1566\ W}{V}$$

Where: A = Water equivalent of the Karl Fischer reagent, gm/ml.
W = Weight of $Na_2C_4H_4O_6 \cdot 2H_2O$ taken, gm.
V = Volume of Karl Fischer reagent used, ml.

(b) Water-in-methanol solution. The water-methanol solution should contain 0.0015-0.0020 gm of water per ml of solution. A good grade of commercial absolute methanol contains about 0.0010 gm water per ml of methanol. Water content can be adjusted by adding 1.0 gm of water to 1000 ml of the water-methanol solution to produce a change of 0.0010 gm per ml. Determine the relative strength of the water-methanol solution in terms of Karl Fischer reagent as follows:

1. Put about 50 ml of the anhydrous methanol used in 4.7.5.2 (a) into the titration beaker of the Aquameter. Add a slight excess of Karl Fischer reagent (4.7.5.2 (a)), then back titrate with water-methanol solution (4.7.5.2 (b)). Then run in an additional 5 to 8 ml of Karl Fischer reagent, read to the nearest 0.01 ml, and again back titrate with water-methanol solution (read to the nearest 0.01 ml). Repeat the addition and back titrating steps twice more to provide triplicate determinations of the equivalency ratio. Calculate the ratio (B) of Karl Fischer reagent to that of the water-methanol solution. The range of the ratios calculated from the three titrations should not be greater than 0.04. If the range

exceeds 0.04, continue making titrations until three ratios are obtained whose range does not exceed 0.04. Then determine the average ratio from all the ratios which have been obtained.

4.7.5.3 Procedure. Determine the moisture content of the sample by the Karl Fischer method using a direct-titration technique. Introduce to the titration beaker, through the opening in the diaphragm, approximately 10 gm of sample weighed to the nearest 0.001 gm and a mixture of 150 ml of anhydrous methanol with 50 ml of glacial acetic acid. Both materials are to be reagent grade. Close the opening, start the stirrer and press the titration button to titrate with Karl Fischer reagent (4.7.5.2 (a)). When the indicator light glows, read the Karl Fischer buret to the nearest 0.01 ml. Where necessary, water methanol solution (4.7.5.2 (b)) may be used to back titrate.

$$\text{Percent moisture} = \frac{100A(V_{KF} - BV_{WM})}{W}$$

Where:
- A = Weight of water equivalent to 1.00 ml of Karl Fischer reagent, gm/ml.
- V_{KF} = Volume of Karl Fischer reagent titrant used, ml.
- V_{WM} = Volume of water-methanol solution titrant used, ml.
- B = Ratio of Karl Fischer reagent to that of water-methanol solution, ml/ml.
- W = Weight of sample taken, gm.

4.7.5.4 Acceptance criteria. For the lot represented to pass the moisture test, the value obtained for the percent moisture shall be no greater than the value specified in 4.3.1.

4.8 Packing and marking. Determine that all packing and marking conforms to section 5 of this document.

5. PREPARATION FOR DELIVERY.

5.1 Preservation and packaging. Not applicable (unless specified in the contract or purchase order).

5.2 Packing.

5.2.1 Level A. Not applicable.

5.2.2 Level B. Not applicable.

5.2.3 Level C. The material shall be packed as directed in the contract to afford protection against damage during direct shipment from the supply source to the first receiving activity for immediate use. Containers shall comply with common carrier regulations applicable to the mode of transportation to be used. (See 6.2.)

5.3 Marking. In addition to the markings required by contract or purchase order, unit packages and shipping containers shall be marked in accordance with the requirements of MIL-STD-129.

6. NOTES.

6.1 Intended use. N-phenylmorpholine described in this document is intended for use as a stabilizer in ammonium-nitrate-based solid propellants.

6.2 Ordering data. Procurement documents should specify the following:

(a) Title, number and date of this document.

(b) Whether a preproduction sample is required (see 3.1).

(c) Type and size of shipping container (see 5.2.3).

6.3 Definition.

6.3.1 Batch. A batch is defined as that quantity of material which has been subjected to one or more chemical or physical processes (or combinations thereof) intended to produce a desired product having substantially uniform characteristics. The final step in the processing must have treated the entire contents of the batch at one time.

6.4 Acceptable product. An acceptable product under this document is N-phenylmorpholine manufactured by the Union Carbide Corporation, Chicago, Illinois.

Custodian:
NASC 52021E

Preparing Activity:
NWC/China Lake, California