MIL-N-3399B
14 MAY 1962
SUPERSEDING
MIL-N-3399A
16 JANUARY 1958

MILITARY SPECIFICATION

2-NITRODIPHENYLAMINE

This specification has been approved by the Department of Defense and is mandatory for use by the Departments of the Army, the Navy, and the Air Force.

1. SCOPE

1.1 This specification covers one type of 2-Nitrodiphenylamine suitable for use in the manufacture of propellants.

1.2 Classification. The 2-Nitrodiphenylamine shall be in the following forms as specified (see 6.1):

Flake form

Powder form

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

UU-S-48 — Sacks, Shipping, Paper.

UU-P-236 — Paper, Filtering.

PPP-B-621 — Boxes, Wood, Nailed and Lock-Corner.

PPP-B-636 — Boxes, Fiber.

STANDARDS

MILITARY

MIL-STD-105 — Sampling Procedures and Tables for Inspection by Attributes.

MIL-STD-109 — Inspection Terms and Definitions.

MIL-STD-129 — Marking for Shipment and Storage.

MIL-STD-286 — Propellants, Sampling, Inspection and Testing.

PUBLICATIONS

ORDNANCE CORPS

ORD-M-608-11 — Procedures and Tables for Continuous Sampling by Attributes.

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

FSC 6810

2.2 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 shall apply.

CONSOLIDATED CLASSIFICATION COMMITTEE PUBLICATION

Consolidated Freight Classification Ratings, Rules, and Regulations.

(Application for copies should be addressed to Consolidated Classification Committee, 202 Chicago Union Station, Chicago 6, Illinois)

AMERICAN TRUCKING ASSOCIATION

National Motor Freight Classification, Rules and Container Regulations.

(Application for copies should be addressed to the National Classification Board, 1424 Sixteenth Street, N. W., Washington 6, D. C.)

3. REQUIREMENTS

3.1 **Chemical and physical properties.** The chemical and physical properties of the 2-nitrodiphenylamine shall conform to the limits specified in table I.

TABLE I. *Chemical and Physical Properties*

Tests	Requirements
Solidification point, Degree Centigrade (°C), minimum (min.)	73.0
Purity, percent, (min.)	97.5
Volatile matter, percent, maximum (max.)	0.6
Material insoluble in 95 percent ethyl alcohol, percent, max.	0.2
Ash, percent, max.	0.1
pH Value	5.0 to 7.0
Acidity, percent, max.	0.003

3.2 **Workmanship.** The standard of workmanship shall be such as to insure the production of material meeting the requirements of this specification. The 2-nitrodiphenylamine shall be free from grit and extraneous foreign material such as sticks, straws, sand and other visible impurities.

4. QUALITY ASSURANCE PROVISIONS

4.1 **General quality assurance provisions.** The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. 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. Reference shall be made to Standard MIL–STD–109 in order to define the terms used herein. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.

4.1.1 *Contractor quality assurance system.* If the contractor desires to utilize a quality assurance system, which is at variance with the quality assurance provisions of 4.2 and 4.3 and other documents referenced herein, he shall submit a written description of the system to the contracting officer for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections to be performed, inspection stations, sampling procedures, methods of inspection, (measuring and testing equipment), and provisions for control and disposition of non-conforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance provisions required by the provisions of 4.2 and 4.3 and the other documents referenced herein. The contractor shall not be restricted to the inspection station or the method of inspection listed in this specification provided that an equivalent control is included in the approved quality assurance procedure. In cases of dispute as to whether certain procedures of the contractor's system provide equal assurance, the comparable procedure of this specification shall apply. The contractor shall

notify the Government of, and obtain approval for, any changes to the written procedure that affects the degree of assurance required by this specification or other documents referenced herein.

4.1.2 *Submission of product.* At the time the completed lot of product is submitted to the Government for acceptance, the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product submitted:

(a) A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.

(b) Quantity of product inspected.

(c) Results obtained for all inspection performed.

(d) Specification number and date, together with an identification and date of changes.

(e) Certificates of analysis on all material procured directly by the contractor when such material is covered by referenced Government specification.

(f) Quantity of product in the lot.

(g) Date submitted.

The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

4.1.3 *Government verification.* Using the contractor's written quality assurance procedure (see 4.1.1), this detailed specification and other contractual documents as a guide the Government inspector shall verify that quality assurance operations performed by the contractor. Verification shall be in accordance with a or b as applicable, the decision being the responsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all quality assurance provisions of other specifications referenced in any of the contractual documents have been complied with. Deviations from prescribed or agreed upon procedures discovered by the Government inspector shall be brought to the attention of the supplier. Disposition of the product and remedial action shall be as directed by the Government inspector and, depending on the nature of the deviation, may consist of lot rejection, screening, re-sampling, re-instruction of the supplier's employees, or other appropriate action:

(a) Verification at the point of manufacture shall be accomplished at unscheduled intervals in accordance with 4.1.3.1 and 4.1.3.2.

(b) Verification at the point of delivery shall be in accordance with 4.1.3.2.

4.1.3.1 *Surveillance.* Surveillance shall include, but is not limited to:

(a) Observation of procedures concerning lot formation and identification.

(b) Observation of sampling procedures and application of acceptance criteria.

(c) Determination that all required examinations and tests are performed in accordance with the prescribed procedures of this specification, or approved equivalents thereto.

(d) Review of procedures for control and disposition of non-conforming material.

4.1.3.2 *Product inspection.* Product inspection shall consist of Government inspection of product which has been previously inspected by the contractor and found to meet the quality assurance provisions of this specification. The inspection by the Government shall be performed in order to determine that the product is of the quality required by this specification and that the contractor's records are reliable.

4.2 Inspection provisions.

4.2.1 *Lot formation.* A lot shall consist of 2-Nitrodiphenylamine from one or more batches and from one manufacturer, in accordance with the same specification, or same specification revision, under one set of operating conditions. Each batch shall consist of that quantity of 2-Nitrodiphenylamine that has been subjected to the same unit chemical or physical process intended to make the final product homogeneous.

4.2.2 *Examination.* Sampling plans and procedures of the following classification of defects shall be in accordance with Standard MIL-STD-105. Continuous sampling plans, in accordance with Handbook ORD-M608-11 may be used if approved by the procuring activity.

4.2.2.1 *Wooden box or fiberboard carton prior to closing (see 5.2.1 or 5.2.2).*

Categories	Defects	Method of inspection
Critical: None defined.		
Major:	AQL 0.65 percent	
	101. Liner pierced or torn	Visual
	102. Liner improperly closed	Visual
Minor:	AQL 1.00 percent	
	201. Type of liner incorrect	Visual

4.2.2.2 *Sealed wooden packing box (see 5.2.1).*

Categories	Defects	Method of inspection
Critical: None defined.		
Major:	AQL 1.50 percent	
	101. Gross weight, (max.)	Approved scale
	102. Box damaged	Visual
	103. Top improperly assembled	Visual
Major:		
	104. Marking (identification) incorrect	Visual
	105. Strapping missing, broken or loose	Visual-Manual
Minor:	AQL 4.00 percent	
	201. Nail protruding	Visual
	202. Marking (identification) missing or illegible	Visual
	203. DOD symbol missing or illegible	Visual
	204. Strapping improperly assembled	Visual-Manual

4.2.2.3 *Sealed fiberboard carton (see 5.2.2).*

Categories	Defects	Method of inspection
Critical: None defined.		
Major:	AQL 1.00 percent	
	101. Gross weight, max.	Approved scale
	102. Assembly torn or pierced	Visual
	103. Marking (identification) incorrect	Visual
	104. Strapping missing, broken or loose	Visual-Manual

Minor:	AQL 4.00 percent	
201.	Stitches missing or loose	Visual
202.	Marking (identification) missing or illegible	Visual
203.	DOD symbol missing or illegible	Visual
204.	Strapping improperly assembled	Visual-Manual

4.2.3 *Testing.*

4.2.3.1 *Sampling by lot.* A random sample of 10 containers shall be selected from each lot. When lots are comprised of 10 containers or less, each container shall be sampled.

4.2.3.2 *Preparation of composite.* A 1.0 ± .2 ounce primary sample of 2-nitrodiphenylamine shall be removed from each of the ten containers in order to equal ten ounces. If there are less than 10 containers, equal primary samples in sufficient quantity to equal 10 ounces, shall be removed from each container. The individual primary samples shall then be combined in order to form a homogeneous composite sample of 10 ounces and subjected to the tests specified in 4.3. If the composite sample fails to comply with any of the requirements specified, the lot shall be rejected.

4.3 Test methods and procedures.

4.3.1 *Determination of solidification point.* The determination shall be conducted as follows: Melt a specimen of approximately 50 grams (gm.) in a covered 250 milliliter (ml.) beaker by immersing the beaker in a water bath at 90° to 95° C. for 15 minutes. Fill the inner tube of the solidification point apparatus shown on figure 1 to within one and one-half inches of the top with the molten 2-nitrodiphenylamine. Insert the stirrer and thermometer in the tube and place the tube and contents in a water bath at 85° to 90° C. for five minutes. Remove the tube from the water bath and place in the apparatus. Stir the molten material constantly making approximately one up and down motion of the stirrer per second. Using a 76 millimeter partial immersion thermometer number American Standard Testing Method 93 C. with a range of 60° — 90° C. record the temperature of the molten material every 30 seconds until solidification begins as indicated by the temperature of the samples rising after reaching a minimum. Continue the stirring until the temperature reaches a maximum.

4.3.2 *Determination of purity.* Either of the following methods may be used. In case of doubt or dispute, the titanous chloride buffer method shall be used.

4.3.2.1 *Titanous chloride buffer method.* The 2-nitrodiphenylamine shall be determined in accordance with Standard MIL STD-286, Method 218.2 except a specimen weighing .20 ± 0.01 gm., shall be quantitatively transferred to 500 ml. carbon dioxide titration flask in paragraph 5.6.

4.3.2.2 *Bromination method.* The 2-nitrodiphenylamine shall be determined in accordance with Standard MIL-STD-286, Method 218.1 (starting at paragraph 5.6) except an accurately weighed specimen of approximately 0.085 to 0.10 gm. shall be transferred to a 250 ml. glass stoppered iodine flask, 25 ml of chloroform shall be added and the sample allowed to dissolve. Sixty ml. of glacial acetic acid followed by exactly 10 ml. of 0.5 Normal (N) potassium bromide-bromate solution shall be added.

4.3.3 *Determination of volatile matter.* The determination shall be conducted as follows: Transfer an accurately weighed specimen of approximately 10 gm. to an evaporating dish having a capacity of about 30 ml. and a diameter of approximately 60 millimeters (mm.). Accurately weigh the dish and contents, then place in an oven at 100° to 105° C. for 3 hours. Cool the dish and contents in a desiccator and reweigh. Calculate the loss in weight as percent volatile matter in the specimen.

4.3.4 *Determination of material insoluble in 95 percent ethyl alcohol.* The determination shall be conducted as follows: Transfer a weighed specimen of about 10 gm. to a 250 ml. beaker. Add 200 ml. of approximately 95 percent ethyl alcohol and stir until the 2-nitrodiphenylamine dissolves. Heat on a steam bath if necessary in order to aid solution of the specimen. Filter the solution through a tared filtering crucible, being careful to transfer any insoluble residue to the crucible by means of a stream of alcohol. Wash crucible and contents five times with 10 ml. portions of hot alcohol. Dry the crucible and contents in an oven at 100° to 105° C. for ½ hour; cool in a desiccator and weigh. Calculate the gain in weight as percent material insoluble in 95 percent ethyl alcohol. In case of an abnormally high result, run a blank on the alcohol used.

4.3.5 *Determination of ash.* The determination shall be conducted as follows: Transfer a weighed specimen of approximately ten gm. to a tared porcelain or silica crucible and add sufficient alcohol to cover the specimen. Place the dish and contents on a hot plate at low heat under a hood and ignite the alcohol. Allow the alcohol and sample to burn until the specimen is completely carbonized and the flame is extinguished. Ignite the crucible over the flame of a Bunsen burner, or in a muffle furnace, until all the carbonaceous matter has been removed; cool in a desiccator and weigh. Calculate the gain in weight as percent ash. In case of an abnormally high result, run a blank on the alcohol used.

4.3.6 *Determination of acidity.* The determination shall be conducted as follows: Transfer a weighed specimen of approximately 10 gm. to a 125 ml. Erlenmeyer flask. Add 50 ml. of boiling distilled water to melt the specimen. Use distilled water having a pH of 6.0 ± 0.5 at 25° C. Close the flask with a rubber stopper and shake the flask and contents vigorously until the 2-nitrodiphenylamine solidifies. Filter the mixture as rapidly as possible by means of a dry filter paper conforming to type II, class 5 of Specification UU-P-236 and immediately determine the pH of the Filtrate at 25° C. by means of pH meter using a glass calomel electrode pair. Wash the residue in the flask twice with 25 ml. portions of distilled water and filter by means of the same filter paper previously used. Combine the washwater with the original filtrate, add 5 drops of phenolphthalein and titrate to a pink end point with 0.01 N sodium or potassium hydroxide solution. Calculate the acidity as follows:

$$\text{Percent acidity (as hydrochloric acid)} \frac{3.65\ VN}{W}$$

where:

V = ml. of sodium or potassium hydroxide solution used in the titration.

N = normality of sodium or potassium hydroxide solution.

W = gm. of the specimen.

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging. Preservation and packaging for levels A, B, and C. Not applicable.

5.2 Packing.

5.2.1 *Level A.* Not more than 100 pounds of 2-nitrodiphenylamine shall be packed in a nailed wooden box conforming to the requirements of Specification PPP-B-621. Each box shall be lined with a single wall bag liner complying with type I, class C, Specification UU-S-48. Adhesive and construction of seams and closures shall be as required for ocean shipment. Boxes shall be strapped in accordance with the strapping requirements of the appendix of Specification PPP-B-621.

5.2.2 *Level B.* The 2 nitrodiphenylamine shall be packed in a solid fiberboard carton conforming to the requirements of V3S, style FTC, Specification PPP-B-636 and shall contain not more than 70 pounds. Each carton shall be lined with a single wall bag liner complying with type I, class B, Specification UU-S-48; adhesive and construction of seams and closures shall be as required for ocean shipment. Cartons shall be strapped in accordance with the strapping requirements of the appendix of Specification PPP-B-636 (see 6.2).

5.2.3 *Level C.* The 2-nitrodiphenylamine shall be packed for shipment in conformance with consolidated freight classification rules and container specifications for rail shipment, or with National Motor Freight classification rules and container specifications for truck shipments.

5.3 Marking. Marking shall be in accordance with Standard MIL-STD-129.

6. NOTES

6.1 Intended use. Flake form and powder form 2-nitrodiphenylamine under this specification are intended for use in the manufacture of propellants.

6.2 Ordering data. Procurement documents should specify the following:

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

(b) Form require (see 1.2).

(c) Levels of protection (see 5.2).

(Level B Packaging is intended to provide economical but limited protection, and should be specified only when it is determined the 2-Nitrodiphenylamine will be held in covered storage approximately 6 months from date of initial packaging.)

Custodians:
Army—Ordnance Corps
Navy—Bureau of Naval Weapons

Preparing activity:
Army—Ordnance Corps

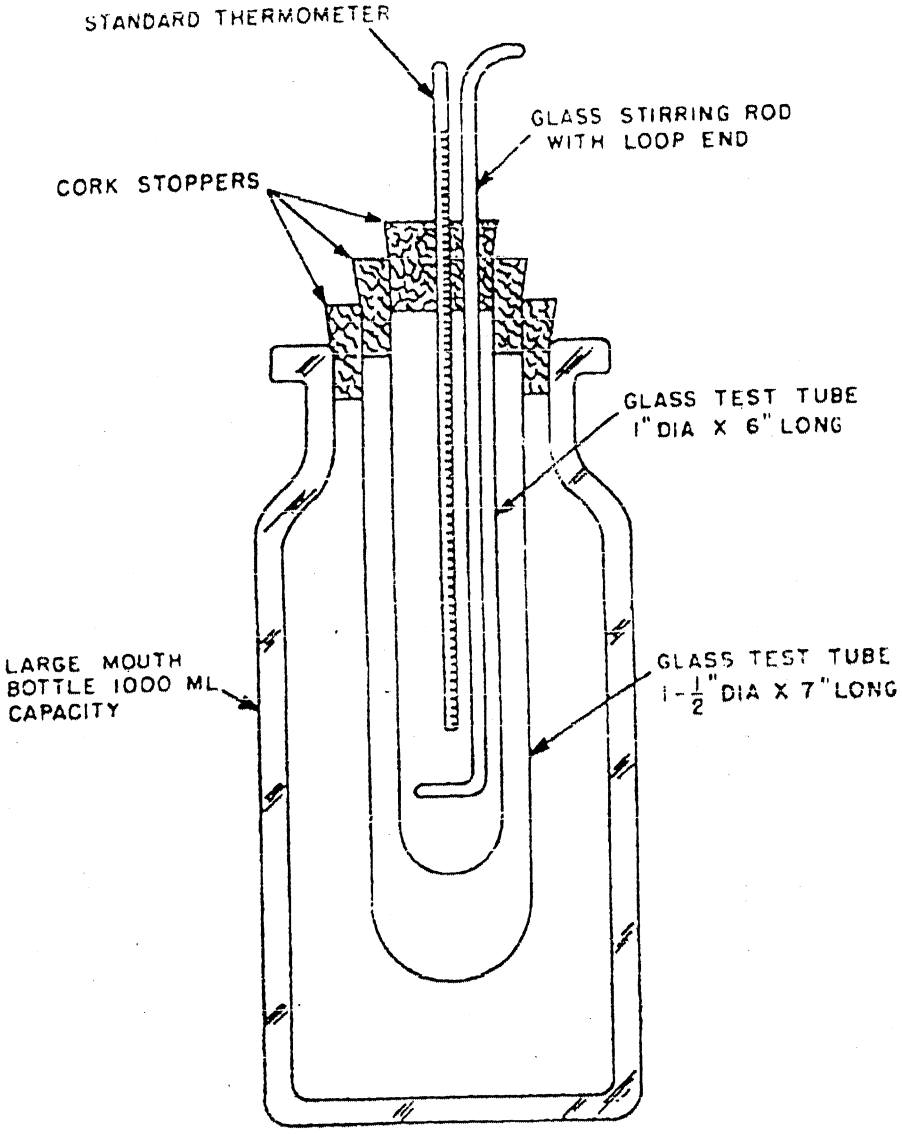

FIGURE 1. *Solidification point apparatus.*

आयुध निर्माणी भंडारा
म्यूनिशन्स इंडिया लिमिटेड
कीड़ीकाई
भारत सरकार का उद्यम
रक्षा मंत्रालय
भंडारा, महाराष्ट्र–441 906.

Ordnance Factory
Bhandara
Unit of Munitions India Ltd.
Govt. of India Enterprise,
Ministry of Defence
Bhandara, Maharashtra– 441 906.

दूरभाष सं/PHONE No. 07184-275742-49 Fax : 07184-275242 email:ofba@ord.gov.in

Quality Assurance Plan

ITEM: 2-NITRO DIPHENYL AMINE

SPECIFICATION/DRAWING: AS PER ATTACHED SPECIFICATION MIL-N-3399B DATE: 14.05.1962 OR IND/ME/877:2015, DC-5401-ME DATE: 01.02.2016.

1. Supplier/ Manufacturer has to submit latest Pre-inspection report/test results from NABL accredited laboratory/ Govt. laboratory for all test requirement as mentioned in above said specification/Supply Order.
2. Firm has to supply the bulk material strictly as per specification/drawing & supply order.
3. Bulk quantity, of the store will be inspected for the following parameter-
   Visual inspection- (Quantity 100%) item should be free from any visual defects/damage.

Dimensional Inspection-as per specification/drawing mentioned above & in the Supply Order.

4. All parameters should be as per above mentioned specification/drawing.
5. All other criteria should be as per the specification/drawing mentioned above.
6. Bulk will be accepted on the basis of inspection & test report of NABL accredited laboratory/Govt. laboratory/OFBa laboratory as per specification/Drawing mention above and all other criteria mentioned in supply order/"any attached sheet" with the supply order should be followed strictly.
7. Item should be appropriately packed to ensure that there is no transit damage.
8. If any latest version of the specification/Drawing mention above is found available and included in the supply order then the latest version will be followed.
9. Clearance of advance sample: Firm has to supply advance sample -
   a. When an item is being developed for the first time.
   b. When an item is being developed through new source.
   c. When an item is being supplied by the firm after laps of three years after completion of last Order.

(D.M. SHENDE)
Divisional Officer
QC(Process)
For General Manager