MILITARY SPECIFICATION

ANTIOXIDANT,

N-PHENYL-N'-CYCLOHEXYL-p-PHENYLENE DIAMINE

This specification is approved for use by the Naval Air Systems Command, Department of the Navy, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE.

1.1 Scope. This specification establishes the requirements for N-phenyl-N'-cyclohexyl-p-phenylene diamine antioxidant material.

2. APPLICABLE DOCUMENTS.

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

STANDARDS

MILITARY

MIL-STD-129	Marking for Shipment and Storage.
MIL-STD-1218	ACS Chemicals.

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

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Air Engineering Center, Engineering Specifications and Standards Department (ESSD), Code 93, Lakehurst, NJ 08733, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 6810

3. REQUIREMENTS.

3.1 Chemical and physical properties. Chemical and physical properties of the material shall conform to Table I.

TABLE I. Chemical and physical properties.

Property	Min	Max
Melting point, °C	110	---
p-Aminodiphenylamine, percent	---	5.0
Diphenylamine, percent	---	0.5

3.2 Stability. When stored at 27 ±11 degrees Celsius (°C), the material shall have a storage life of 24 months from date of delivery to the procuring activity.

3.2.1 Storage life extension. The storage life of an individual lot (see 6.3.1) of material may be extended an additional 12 months provided the material, upon retest, successfully meets the requirements for melting point, p-aminodiphenylamine, and diphenylamine.

3.3 Infrared (IR) spectrum. An IR spectrum shall be made. The IR spectrum shall qualitatively conform to Figure 1.

3.4 Toxic products and safety. Safety regulations and guidelines applicable to the use of N-phenyl-N'-cyclohexyl-p-phenylene diamine should be complied with to preclude personal injury and damage to equipment and facilities.

3.5 Workmanship. Workmanship shall be such that the material is uniform in appearance and free from visible contamination.

4. QUALITY ASSURANCE PROVISIONS.

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order (see 6.2.1), the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the contractor 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 specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Sampling. The lot shall be sampled in accordance with Table II.

TABLE II. Sampling plan.

Number of containers in lot	Number of containers sampled (primary sample)	Number of composite samples
100 or more	10% (nearest whole number)	5
51 - 99	10	4
11 - 50	10	3
1 - 10	ALL	2

4.2.1 Primary samples. Physical properties tests shall be run on each primary sample (see Table II). The material container may be sampled by use of a clean glass tube, rod, or pipet. If the container is small enough to be handled safely, the sample may be obtained by pouring. The smallest sample size possible that is consistent with test requirements shall be taken. The minimum sample size shall be two ounces. Glass containers shall be used for all liquid samples. Each sample shall be labeled with date, lot number, and manufacturer's container identification number. Failure of any primary sample to pass all of the physical properties tests herein shall result in rejection of the lot represented.

4.2.2 Composite samples. Chemical properties tests shall be run on each composite sample. Divide the primary samples equally into the number of composites shown in Table II. Blend each composite thoroughly by manipulation of the container. Label each composite with Roman numerals, also include date, lot numbers, and manufacturer's container identification numbers. The remainder of the primary samples shall be retained pending acceptance or rejection of the lot. Failure of any composite sample to pass all of the chemical properties tests herein shall result in rejection of the lot represented.

4.3 Quality conformance inspections and tests. Quality conformance inspections and tests shall consist of the following:

a. Tests of Table I properties (see 4.4).

b. Inspection of filled containers (see 4.5.1).

c. Visual inspection (see 4.5.2).

4.4 Test methods. Tests shall be performed using apparatus, reagents, and procedures specified herein. The use of alternate apparatus, reagents, or procedures shall require prior written approval of the procuring activity. All American Chemical Society (ACS) reagents shall conform to MIL-STD-1218.

4.4.1 Melting point.

4.4.1.1 Apparatus.

a. Melting point apparatus, filled with mineral oil, or equal.

b. Melting point capillary tubes, 100 millimeter (mm) by 1.5 to 2.0 mm.

c. Thermometer, 0 to 150°C, or equal.

4.4.1.2 Determination of melting point. Collect a small amount of sample in the open end of a capillary melting point tube. Pack sample into about 3 mm of the capillary tube. Preheat the bath to within 3°C of the expected melting point. Attach the capillary tube to the thermometer with the sample opposite the mercury bulb and lower the assembly into the melting point apparatus until the thermometer bulb is below the side arm. The liquid level of the bath should be near the 0°C mark when melting occurs. Adjust heating so that the temperature rise is approximately 1°C per minute when approaching the melting point. Record the temperature when the sample becomes completely wet and molten. Report the melting point to the nearest °C.

4.4.2 p-Aminodiphenylamine.

4.4.2.1 Apparatus.

a. Spectrophotometer, Perkin-Elmer Model 350, or equal, with matched 1 centimeter silica cells, set up to measure in the 450 nanometer (nm) range.

b. Micro buret, 10-milliliter (ml).

4.4.2.2 Reagents.

a. p-Aminodiphenylamine, reagent grade.

b. Glacial acetic acid, ACS reagent.

c. Salicylaldehyde, reagent grade.

4.4.2.3 Preparation of standard p-aminodiphenylamine curve.

NOTE: Standardization must be completed in 1 day and must be repeated for each new lot of glacial acetic acid.

Weigh accurately 0.12 gram (gm) of p-aminodipheylamine into a 100-ml volumetric flask. Dissolve in glacial acetic acid, dilute to volume with glacial acetic acid, and mix well. Transfer, by means of a micro buret, 2.0, 4.0, and 6.0 ml of the solution to a series of 100-ml volumetric flasks. Dilute each to volume with glacial acetic acid and mix well. Prepare two 100-ml volumetric flasks, marked A and B, by transferring 10-ml aliquots from each flask. Add 40 ml of glacial acetic acid and 45 ml of distilled water to each flask and swirl to mix. Fill flask B to volume with distilled water, mix well, and use flask B solution to zero the spectrophotometer. Add 1.0 ml of salicylaldehyde to flask A, dilute to volume with distilled water and mix well. Determine the absorbance of the sample solution immediately (absorbance decreases on standing) at a wave length of 450 nm and a slit width no greater than 0.2 mm. Repeat this procedure for the other three sample solutions. Prepare a standard curve by plotting the absorbance versus the concentration of p-aminodiphenylamine in gm/100 ml, and draw a line best fitted to the points.

4.4.2.4 Determination of p-aminodiphenylamine.

NOTE: Analysis must be completed in 1 day and requires the same lot of glacial acetic acid used for the standard curve.

Weigh accurately 0.60 gm of sample into a 50-ml volumetric flask, dissolve in glacial acetic acid, dilute to volume with glacial acetic acid and mix well. Transfer 10.0 ml of sample solution to a 100-ml volumetric flask, dilute to volume with glacial acetic acid and mix well. Transfer 10.0 ml of this solution into each of two 100-ml volumetric flasks marked A and B. Add 40 ml of glacial acetic acid and 45 ml of distilled water to each flask and swirl to mix. Fill flask B to volume with distilled water, mix well, and use flask B solution to zero the spectrophotometer. Add 1.0 ml of salicylaldehyde to flask A, dilute to volume with distilled water and mix well. Determine the absorbance of the sample solution immediately (absorbance decreases on standing) at a wave length of 450 nm and a slit no greater than 0.2 mm. Read the concentration of p-aminodiphenylamine in the sample solution directly from the standard curve.

Calculation:

$$\text{p-aminodiphenylamine, percent} = \frac{C \times 500}{W}$$

Where: C = concentration from standard curve, gm/100 ml

W = weight of sample, gm

Report the p-aminodiphenylamine to the nearest 0.1 percent.

4.4.3 Diphenylamine.

4.4.3.1 Apparatus.

a. Gas chromatograph, Perkin-Elmer Model 820, equipped with a 12 foot stainless steel column by 0.12-inch outside diameter, packed with 15 percent W/W Apiezon L on Anakrom ABS, 90 to 100 mesh, or their equal.

b. Helium cylinder.

c. Syringe, 5-microliter.

4.4.3.2 Reagents.

a. Diphenylamine, ACS.

b. Acetone, ACS.

4.4.3.3 Preparation of standard diphenylamine solution. Weigh accurately 0.08 gm of diphenylamine into a 100-ml volumetric flask. Dissolve in acetone, dilute to volume with acetone and mix well. Inject 5 microliters of this solution into the gas chromatograph using the following conditions:

Injection port	275 to 300°C
Column temperature	250°C
Detector	275 to 300°C
He flow	40 cubic centimeters/minute

Calculate the standard factor from the peak height thus obtained.

Calculation:

$$\text{Standard factor} = \frac{W}{100 \times P}$$

Where: W = weight of sample, grams

P = peak height of sample, mm

Record the standard factor to the nearest 0.000001.

4.4.3.4 Determination of diphenylamine. Weigh accurately 4.0 gram of sample into a 25-ml volumetric flask. Dissolve in acetone, dilute to volume with acetone and mix well. Inject 5 microliters of sample solution into the gas chromatograph which has been set to the conditions including attenuation in 4.4.3.3. Calculate the amount of diphenylamine in the sample from the peak height thus obtained.

Calculation:

$$\text{Diphenylamine, percent} = \frac{F \times P \times V \times 100}{W}$$

Where: F = standard factor from 4.4.3.3

P = peak height of sample, mm

V = volume of sample solution, ml

W = weight of sample, gm

Report the diphenylamine to the nearest 0.1 percent.

4.4.4 Infrared spectrum. The infrared spectrum shall be determined using an infrared spectrophotometer in accordance with the following:

a. Set the instrument pen to at least 70 percent transmittance at approximately 4,000 cm^{-1} when a pellet of spectrographic grade potassium bromide and N-Phenyl-N'-Cyclohexyl-p-Phenylene Diamine (approximately 55:1 weight ratio) is mounted in the sample beam (reference beam:air).

b. Adjust the thickness of the pellet if 70 percent transmittance is not achieved. Also, adjust the weight ratio of the pellet if any major peaks are less than two percent transmittance.

c. Scan the pellet and compare resulting spectrum to Figure 1. The peak position and relative magnitude shall be qualitatively similar within a 2.5 to 15 µm, scan range.

d. Report conformance to, or deviation from, Figure 1.

4.5 Examinations.

4.5.1 Inspection of filled containers. All filled containers shall be inspected prior to shipment or use for accuracy of markings and for

defects in containers and closures. All defective containers and closures shall be repaired or replaced, and contents therein shall be reinspected prior to shipment or use.

4.5.2 Visual inspection. All samples shall be visually inspected to determine conformance to the requirements of 3.5.

4.6 Records. Certification and test data shall be prepared as required by the procuring activity (see 6.2.2).

5. PACKAGING.

5.1 Packaging and packing. Unless otherwise specified in the contract or purchase order (see 6.2.1), packaging and packing of the material shall be in accordance with commercial practice to insure carrier acceptance and shall be of such construction and materials that the contents will be adequately protected against loss or contamination.

5.2 Marking for shipment. Unless otherwise specified in the purchase order (see 6.2.1), each shipping container shall be marked in accordance with the requirements of MIL-STD-129. Container marking shall include the following:

a. The supplier's lot number.

b. Procuring activity purchase order number.

c. Container identification number (applied in numerical sequence as the containers are filled).

d. Date of manufacture.

e. Manufacturers' Code Ident.

f. Net and tare weight of container.

g. Material identification.

6. NOTES AND CONCLUDING MATERIAL.

6.1 Intended use. The intended use of the material described herein is as an antioxidant in solid rocket motor propellant.

6.2 Ordering data.

6.2.1 Procurement requirements. Procurement documents should specify the following:

a. Title, number and date of this specification.

b. Responsibility for inspection and inspection facilities, if different than 4.1.

c. Special packaging, packing, or shipping requirements, if applicable (see Section 5).

6.2.2 Data requirements. When this specification is used in a procurement which incorporates a Contract Data Requirements List (DD Form 1423) and invokes the provisions of 7-104.9(n) of the Defense Acquisition Regulations (DAR), the data requirements identified below will be developed as specified by an approved Data Item Description (DID) (DD Form 1664) and delivered in accordance with the approved DD Form 1423 incorporated into the contract. When the provisions of DAR-7-104.9(n) are not invoked, the data specified below will be delivered by the contractor in accordance with the contract requirements. Deliverable data required by this specification is cited in the following paragraphs:

Paragraph	Data Requirement	Applicable DID
4.6	Certification Test Data	UDI-A-23264B DI-T-4024

(Copies of DIDs required by the contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

6.3 Definitions.

6.3.1 Lot. At place of manufacture, a lot consists of one batch (see 6.3.2) or a uniform blend of two or more batches. At place of delivery, a lot consists of material from one supplier's lot received in a single shipment. Partial shipments may be considered as a single shipment by the procuring activity.

6.3.2 Batch. A batch consists of material made as one unit in an unchanged manufacturing process.

6.4 Suggested source of supply. A product that has met the requirements of this specification in past procurement actions is Flexone® 6H manufactured by Uniroyal Chemical Division of Uniroyal, Incorporated, Code Ident 80196. This information is for the convenience of the procuring activity and is not to be construed as a waiver of any requirement of this specification nor as any limitation of additional potential sources of supply.

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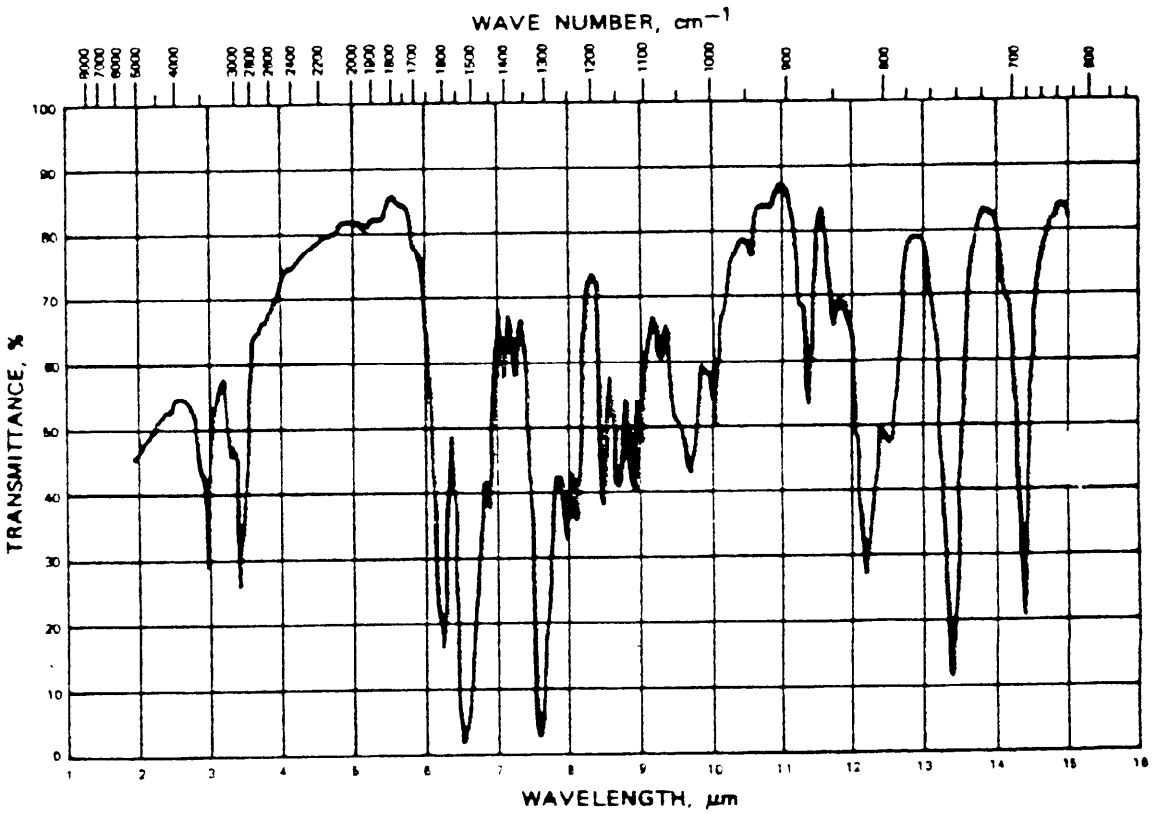

Figure 1. Typical infrared spectrum.

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