

MIL-A-85495(AS)
6 October 1981

MILITARY SPECIFICATION

ANTIOXIDANT, T-BUTYLPHENOL TYPE, 2, 2'-METHYLENEBIS

(4-METHYL-6-t-BUTYLPHENOL)

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 an antioxidant of the t-butylphenol type, 2, 2'-methylenebis (4-methyl-6-t-butylphenol).

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.

SPECIFICATIONS

MILITARY

MIL-C-85498	Curing Agents, Dimeryl-di-isocyanate and Isophorone di-isocyanate.
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STANDARDS

MILITARY

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

DRAWINGS

Naval Air Systems Command
(Code Ident 30003)

639AS4616	Polybutadiene Polymer.
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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.

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

3. REQUIREMENTS.

3.1 Product characteristics. The antioxidant covered by this specification shall be a pale cream to white color, and shall be a crystalline powdery solid.

3.2 Chemical and physical properties. The chemical and physical properties of the antioxidant shall conform to Table I.

TABLE I. Chemical and physical properties.

Property	Min	Max
Isocyanate (NCO) uptake, (equivalent NCO/grams antioxidant)	---	.005
Melting point range, (°C)	120	130

3.3 Toxic products and safety. Safety regulations and guidelines applicable to the use of antioxidant should be complied with to preclude personal injury and damage to equipment and facilities.

3.4 Workmanship. Workmanship shall be such that the antioxidant is uniform, of consistent high quality, and free from 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 shall be 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.

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

- a. Test 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.

4.4.1 NCO uptake.

4.4.1.1 Apparatus.

- a. Infrared spectrophotometer, Perkin-Elmer Model 21, equipped with 1.0 millimeter (mm) sodium chloride (NaCl) cells, or equal.
- b. Absorbance chart paper, Perkin-Elmer No. 021-6313, or equal.
- c. Snap cap bottles, Wheaton, 6 or 10 dram, or equal.

4.4.1.2 Reagents.

- a. Polybutadine polymer (phenyl-beta-naphthylamine removed) (Drawing 639AS4616).
- b. Isophorone di-isocyanate (IPDI) in accordance with MIL-C-85498, Type II.
- c. Carbon tetrachloride, spectrophotometry grade in accordance with MIL-STD-1218.

4.4.1.3 Determination of NCO uptake. Weigh accurately 0.1000 gram (gm) of sample, 2.5 gm of polybutadine polymer and 0.184 gm of IPDI in a snap cap bottle. Mix well and weigh accurately 0.35 gm of this solution into a 20-milliliter (ml) beaker. Dissolve in a few ml of carbon tetrachloride, transfer quantitatively to a 25-ml volumetric flask, dilute to volume with carbon tetrachloride, and mix thoroughly. Prepare a blank in like manner. Measure the infrared absorbance at 2270 cm^{-1} in a 1.0 mm NaCl cell for both the sample and blank. Place cap on both sample and blank solutions remaining in the initial bottle and place in 60 degree Celsius ($^{\circ}\text{C}$) oven for 3 days. Again determine the infrared absorbance of 0.35 gm of sample and blank in a 25-ml volumetric flask with carbon tetrachloride as above. Using the initial and final absorbance values, calculate the amount of NCO which has reacted with the sample.

Calculation:

$$\text{NCO uptake, (equivalent NCO/gm antioxidant)} = \frac{W_1 \times F \times A}{W}$$

Where: W_1 = weight of IPDI, gm

F = 0.8975 equivalent NCO/100 gm IPDI = 0.008975

W = weight of antioxidant, gm

$$A = \frac{(A_1 - A_2)}{A_1} - \frac{(A_3 - A_4)}{A_3}$$

A_1 = absorbance of sample at 0 time

A_2 = absorbance of sample after 3 days

A_3 = absorbance of blank at 0 time

A_4 = absorbance of blank after 3 days

Report the NCO uptake to the nearest 0.0001 equivalent NCO/gm antioxidant.

4.4.2 Melting point range.

4.4.2.1 Apparatus.

- a. Melting point apparatus, filled with mineral oil, or equal.
- b. Melting point capillary tubes, 100 mm by 1.5 to 2.0 mm.
- c. Thermometer, 0 to 150°C, or equal, graduated in 0.1°C.

4.4.2.2 Determination of melting point range. Collect a small amount of sample in the open end of a capillary melting tube. Pack the sample into about 3 mm of the capillary tube. Preheat the batch to within 3°C of the expected melting point. Attach the capillary to the thermometer with the sample adjacent to 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 batch 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 first begins to melt and again when the sample has completely melted. Report the melting point range as these two temperatures to the nearest 0.1°C for each.

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4.5 Examinations.

4.5.1 Inspection of filled containers. All filled containers shall be inspected prior to shipment or use for accuracy or 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 3.4.

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 antioxidant 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 Container marking. Unless otherwise specified in the contract or 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. Manufacturer's Code Ident.
- f. Net and tare weight of the container.
- g. Material identification.

6. NOTES AND CONCLUDING MATERIAL.

6.1 Intended use. The intended use of the material described herein is to prevent oxidation of liquid polymers.

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:

<u>Paragraph</u>	<u>Data Requirement</u>	<u>Applicable DID</u>
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 antioxidant from one manufacturer's batch or blend, or if manufactured by a continuous process, any quantity of material made from homogeneous raw materials and during a period of uninterrupted operations. At place of delivery, a lot consists of antioxidant from one manufacturer's lot received in a single shipment. Partial shipment may be considered a single shipment at the option of the procuring activity.

6.4 Suggested source of supply. A product that has met the requirements of this specification in past procurement actions is marketed by American Cyanamid Company, Code Ident 89457, as Antioxidant 2246R. 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.

Preparing activity:
Navy - AS

(Project 6810-NB21)

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DOCUMENT IDENTIFIER (Number) AND TITLE (MIL-A-85495(AS), ANTIOXIDANT, T-BUTYLPHENOL
TYPE, 2, 2'-METHYLENEBIS (4-METHYL-6-t-BUTYLPHENOL)

NAME OF ORGANIZATION AND ADDRESS OF SUBMITTER

VENDOR USER MANUFACTURER

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C. REASON FOR RECOMMENDED CHANGE(S)

2. REMARKS

SUBMITTED BY (Printed or typed name and address - Optional)

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DD FORM 1426
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