MILITARY SPECIFICATION

4,4' - METHYLENEBIS (2,6-DITERTBUTYLPHENOL)

This specification is approved for use within the US Army Armament, Munitions and Chemical Command and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers the requirements and quality assurance provisions for the manufacture and acceptance of one type of 4,4' Methylenebis (2,6-ditertbutylphenol) (see 6.1).

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. The following specifications and standards form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation.

SPECIFICATIONS

MILITARY

MIL-A-48078 - Ammunition, Standard Quality Assurance Provisions, General Specification for

STANDARDS

MILITARY

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

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, U.S. Army Armament, Munitions and Chemical Command, Attn. AMSMC-QA, Picatinny Arsenal, New Jersey 07806-5000 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A
FSC 6810

MIL-STD-129 - Marking for Storage Shipment
MIL-STD-286 - Propellants, Solid: Sampling, Examination and Testing
MIL-STD-1168 - Lot Numbering of Ammunition
MIL-STD-1218 - ACS Chemicals

(Copies of specifications, standards, handbooks, drawings, publications, and other Government documents required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting activity.)

2.1.2 Other Government documents, drawings, and publications. The following other government documents, drawings, and publications form a part of this specification to the extent specified herein. Unless otherwise specified, the issues shall be those in effect on the date of the solicitation.

CODE OF FEDERAL REGULATIONS
Title 49, Transportation, Parts 100-199

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402. Orders for the above publication should cite: 49 CFR 100-199 (latest revision).

2.2 Other publications. The following document(s) form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted shall be those listed in the issue of the DODISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS shall be the issue of the nongovernment documents which is current on the date of the solicitation.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM E300-73 - Recommended Practice for Sampling Industrial Chemicals

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

UNITED NATIONS

United Nations - Transport of Dangerous Goods (Recommendations of the Committee of Experts on the Transport of Dangerous Goods)

(Application for copies should be addressed to United Nations, Sales Section, First Ave., New York, N.Y.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, (except for associated detail specifications, specification sheets or MS standars) the text of this specification shall take precendence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Material. The 4,4' Methylenebis (2, 6-ditertbutylphenol) shall conform to the following requirements when tested in accordance with the applicable test method.

Property	Min.	Max.	Applicable Test Method
Assay, mole %	98	---	4.5.1
Moisture, wt. %	---	0.5	4.5.2

3.2 Color. The 4,4' Methylenebis (2,6-ditertbutylphenol) shall be a white to light yellow powder, when tested in accordance with 4.5.3.

3.3 First article inspection. This specification contains technical provisions for first article inspection. Requirements for the submission of first article samples by the contractor shall be as specified in the contract.

3.4 Workmanship. The manufacturer shall implement procedures and controls to assure that the process and the product produced are not compromised by foreign materials and contaminants or any other conditions which may degrade the composition. Determination of foreign materials shall be in accordance with 4.5.4.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is

responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor 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.

4.1.1 Responsibility for compliance. All items must meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absense of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling in quality conformance does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to acceptance of defective material.

4.2 Classification of inspections. The inspection requirements specified herein are classified as follows

a. First article inspection (see 4.3).

b. Quality conformance inspection (see 4.4).

4.3 First article inspection.

4.3.1 Submission. The contractor shall submit a first article sample as designated by the Contracting Officer for evaluation in accordance with provisions of 4.3.2. The first article sample shall be ten (10) pounds of 4,4' Methylenebis (2,6-ditertbutyl-phenol). The sample shall be obtained from the first production lot which has been produced by the contractor using the same production processes, procedures and equipment as will be used in fulfilling the contract. All materials shall be obtained from the same sources of supply as will be used in regular production.

4.3.2 Inspection to be performed. The sample will be subjected by the Government to any or all of the examinations or tests specified in Table II (MIL-A-48078).

4.3.3 Rejection. See MIL-A-48078.

TABLE II. First article inspection

CLASSIFICATION OF DEFECTS & TESTS

MIL-M-70691 (AR)

PARAGRAPH	TITLE		SHEET OF		DRAWING NUMBER / NEXT HIGHER ASSEMBLY
CATEGORY	EXAMINATION OR TEST	NO. OF SAMPLE UNITS	AQL OR 100%	REQUIREMENT PARAGRAPH	PARAGRAPH REFERENCE /INSPECTION METHOD
	Assay, mole %			3.1	4.5.1
	Moisture, wt. %			3.1	4.5.2
	Color			3.2	4.5.3
	Workmanship			3.4	4.5.4
NOTES:					

AMSMC Form 1570 1 Feb 85 Replaces DRSMC-DA (D) Form 160 1 Aug 83 which may not be used

4.4 Quality conformance inspection.

4.4.1 Lot formation. A lot shall consist of one or more batches of 4,4' Methylenebis (2,6-Ditertbutylphenol) produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each lot shall consist of that quantity of 4,4' Methylenebis (2,6-Ditertbutylphenol) that has been subjected to the same unit chemical or physical process intended to make the final product homogeneous. The lot shall comply with the provisions for submission of product as specified in MIL-STD-105. The criteria and procedure for the assignment of lot numbers shall be in accordance with MIL-STD-1168. Also, MIL-A-48078 applies.

4.4.2 Examination. See MIL-A-48078.

a. Workmanship. Examination for workmanship shall be conducted on each sample for testing in accordance with 4.4.3.1. If any sample fails to meet any test requirement the batch represented by the sample shall be rejected.

4.4.3 Testing. PRECAUTION: This specification covers sampling and testing of toxic and hazardous materials. Accordingly, it is emphasized that all applicable safety rules, regulations and procedures must be followed in handling and processing the 4,4' Methylenebis (2,6-Ditertbutylphenol).

4.4.3.1 Sampling. Approximately 500 grams of the composition shall be selected from each batch to be sampled using ASTM Method E300-73 for solids. Samples shall be selected for inspection in accordance with table below. If any sample fails to meet any test requirement with batch represented by the sample shall be rejected. Each required test shall be performed in duplicate. No composite samples shall be used. The classification of defects shall be as given in Table III.

Number of containers in batch	Number of containers to be sampled
2 - 100	2
100 - 200	5
200 - 300	8
300 - 500	10
500 - 1000	13
1000 - 3000	20
3000 - 10,000	32

TABLE III. Classification of defects

Test Examination	Paragraph	Defects Classification
Assay	3.1	Major
Moisture	3.1	Major
Color	3.2	Major
Workmanship	3.4	Major

4.4.4 Inspection equipment. For the performance of all tests and examinations specified in 4.4 and 4.5, commercial inspection equipment should be employed. The contractor shall have available and utilize correctly, this equipment and is charged with the responsibility of assuring that proper calibration procedures are followed.

4.5 Methods of inspection. All tests given in this section shall be performed using prescribed procedures for replicate determinations given in standard analytical chemistry textbooks. Also, unless otherwise specified herein, all chemicals shall be Reagent Grade or ACS Grade in accordance with MIL-STD-1218. See 6.3 for use of equivalent test methods.

4.5.1 Assay. Assay shall be determined in accordance with the following:

4.5.1.1 Apparatus. Set-Point Apparatus, comprising a freezing tube, stirrer, thermometers, heating bath and laboratory jack as follows:

a. Freezing Tube, 15 ml capacity double-walled well, fabricated from borosilicate glass.

b. Hot Plate, electric, capable of maintaining oil bath at 165-180°C. Tempco Model HP-19158, Model 1900 (700 watts) is satisfactory.

c. Laboratory Jack, Cenco-Lerner No. 19089 or equivalent.

d. Oil Bath, stainless steel cylinder, approximately 4 inches diameter and 4 inches deep. Can be made by cutting down a 1200 ml beaker.

e. Stirrer, stainless steel wire, approximately 16 guage, with one end coiled into several loops so that the loops will encase the set-point thermometer.

f. Thermometer, total immersion, 150-160°C, 8-10 inches total length, graduated to 0.1°C with lowest graduation at least 3-1/2 inches above tip of thermometer. Available on special order

from Precision Thermometer and Instrument Company, Southhampton Industrial Park, Southhampton, Pennsylvania 18966. Calibrate against NBS certified thermometer.

g. Thermometer, 0-100°C, graduated to 0.1°C.

4.5.1.2 Reagents and materials.

a. Nitrogen

b. Silicone Oil, Dow Corning 200 Fluid, 100 centistrokes viscosity. Dow Corning 550 or 710 fluids may also be used.

4.5.1.3 Determination of stem correction.

a. For the thermometer specified , determine the stem correction initially and use the value found as a constant thereafter. A typical stem correction is 0.18°C. Apply the correction as directed in 4.5.1.5. If a different type of thermometer is used, determine a stem correction for each set-point measurement. Apply the correction as directed in 4.5.1.5.

b. Tape a second thermometer, range 0.100°C, to the set-point thermometer. Align the bulb with the 145°C division of the set-point thermometer. Determine a set-point as described in 4.5.1.4. Immediately after reading the set-point on the set-point thermometer, read the air temperature on the second thermometer. Take this reading as exposed stem. Measure the height of the mercury column of the set-point thermometer above the level of the melt. Express the height in degrees Centigrade. If necessary, measure the length of the mercury column in mm and transpose it to degrees Centigrade by multiplying the length in mm by degrees per mm.

4.5.1.4 Procedure

a. Assemble the set-point apparatus in a location free from drafts. Attach a clamp to the neck of the freezing tube. Clamp the freezing tube to a solid support at a height that will allow the laboratory jack, hot plate and oil bath to be moved under the freezing tube. Clamp the set-point thermometer in position above the freezing tube with a large versatile clamp. Make certain that the apparatus is free from contaminates. Set the hot plate on the laboratory jack. Set the oil bath on the hot plate. Add sufficient silicone oil to the oil bath so that the freezing tube will be immersed up to the side arm.

b. Lower the bath away from the freezing tube and heat it to about 165-180°C.

c. Flush the apparatus with nitrogen and connect the nitrogen line to the side arm of the freezing tube. Maintain a slow flush of nitrogen (about 3 bubbles per sec.) until all set-point operations are complete.

d. Prepare a paper funnel, 10 cm long, to extend 1 cm into the well and fit the well tightly. Cut an opening in the paper to allow passage of the nitrogen flush over the material in the well.

e. Add about 11 gm of the material to be tested to the well through this funnel. Do not remove the funnel as the volume of the material while in the solid state usually exceeds the well capacity. Place the stirring rod on top of the solid.

f. Place the heated oil bath beneath the freezing tube. Raise the oil bath until the freezing tube is immersed up to the outer jacket side arm.

g. As the material in the well begins to melt, gently tap the solid with the stirrer to force it down into the melt. Avoid plunging the stirrer through the solids into the melt. When the sample is melted, place the stirrer in the melt and lower the set-point thermometer within the coil of the stirrer so that the bulb is centrally located within the mass of the melt. Do not let the thermometer touch the apparatus.

h. When the melt temperature reaches 160°C, remove the bath from the apparatus and move it several feet away.

i. As the temperature of the melt reaches about 158°C, stir the melt. Maintain constant regular stirring until the test is completed. When stirring is initiated, the melt temperature will first rise to 163-165°C, and then fall to about 149°C. As the melt solidifies, the temperature will rise to a maximum value, which is taken as the observed set-point. Read the set-point temperature to 0.01°C using a meniscus reader (Note 1).

NOTE 1. The stirring appears to be a major factor in achieving duplicate results. Care must be taken to maintain constant stirring, but the stirring should not be too vigorous as this causes low readings. As the end-point is approached, the material sets up as a hard solid which becomes difficult to agitate. Some practice is required before an operator can obtain satisfactory duplicate results.

The set-point may be repeated on the same sample. The oil bath is moved under the set-point apparatus and raised into position. The solid in the freezing tube well is remelted. Material attached to the sides of the well is pushed into the melt with a clean spatula. Care should be taken not to contaminate the

melt with the spatula. When the melt reaches 160°C, the bath is lowered and removed, and the set-point operation repeated. Results should agree within 0.08°C.

j. After the last determination has been performed on the sample, clean the apparatus as follows: Remelt the sample. Withdraw the thermometer; then withdraw the stirrer. Pour the melt into a waste container. Cool the apparatus to room temperature by blowing air over it. Wash the well, thermometer and stirrer with acetone until no residue of sample or other contaminant can be seen. Swab each part with an acetone-wet cloth and flush it four times with acetone. Dry each part by blowing air across it (Note 2).

NOTE 2. If equipment is not cooled to room temperature, it may break while being washed. Contamination of the well with silicone oil must be avoided, as this material is extremely difficult to remove.

4.5.1.5 Calculations.

a. When a constant stem correction is used, calculate the corrected set-point as follows:

Corrected set-point, °C = $T_1 + C + B$.

Where: T_1 = observer set-point, °C.

C = stem correction.

B = calibration correction factor, if any.

b. When a stem correction is determined with the set-point, calculate the corrected set-point as follows:

Corrected set-point, °C = $T_1 + 1.6 \times 10^{-4} A(T_1 - T_2) + B$.

Where: A = height of mercury column of set-point thermometer, °C, 4.5.1.3.

B = calibration correction factor, if any, °C.

T_1 = observed set-point, °C.

T_2 = observed air temperature, °C.

c. For "Ethyl" Antioxidant 702, obtain mol percent purity by referring the corrected set-point to the graph of "Ethyl" Antioxidant 702 purity as a function of set-point.

4.5.2 Moisture. The moisture content shall be determined in accordance with MIL-STD-286, Method 101.5.

4.5.3 Color. The sample shall be examined for conformance with proper color.

4.5.4 Workmanship. Visually examine the sample for contamination or foreign matter.

5. PACKAGING

5.1 Packaging and Packing.

5.1.1 Level A. - When applicable packaging shall be as specified in the contract.

5.1.2 Level B .- Unless otherwise specified in the contract, packaging and packing shall be in accordance with the standard commercial practice applicable to the type of material. The packaging and packing shall be of such construction and materials that the contents will be adequately protected against damage, loss, or contamination when shipped from the contractor or supply source to the receiving activity, or stored for one year from the date of manufacture. Container size/capacity shall be as specified in the contract. The type of containers and packing materials selected shall comply with applicable carrier rules and regulations. In addition, any hazardous material must be packed and shipped in accordance with the Code of Federal Regulations (CFR), Title 49, Parts 100-199, and also when offered for overseas shipment, the United Nations-Transport of Dangerous Goods (Recommendations of the Committee of Experts on the Transport of Dangerous Goods). It shall be the responsibility of the supplier to obtain all data necessary from DOT, and if applicable, from the Committee of Experts on the Transport of Dangerous Goods (UN-Transport of Dangerous Goods) to ship hazardous materials legally.

5.1.3 Level C. - Same as Level B, except not authorized for overseas shipment and that the United Nations-Transport of Dangerous Goods does not apply.

5.2 Marking. In addition to any special marking required by the contract, each container shall be marked in accordance with MIL-STD-129, the Code of Federal Regulations, (CFR) Title 49, Parts 100-199, and the United Nations-Transport of Dangerous Goods (Recommendations of the Committee of Experts on the Transport of Dangerous Goods). Marking shall include but not be limited to, the following information:

a. Title, Number and Date of this Specification

b. Manufacturer's Name and Location

c. Material Trade Name

d. Net Weight and Volume

e. Lot Number, Batch Number(s), and Date of Manufacture

f. Storage Conditions/Requirements

g. Toxicity Precautions, if applicable

h. Contract Number

i. Warranty Provisions

6. NOTES.

6.1 Intended use. The 4,4' Methylenebis (2,6-ditertbutylphenol) is intended for use in M864 propellant.

6.2 Ordering data. See MIL-A-48078 (PA).

6.3 Equivalent test methods. The test methods given in this specification are the official methods to be used. The contractor may request using other methods providing that the proposed method is equivalent (accuracy and precision) to the method given in this specification. Prior approval of the Contracting Officer is required for use of equivalent test methods. A description of the proposed method should be submitted through the Contracting Officer to: Commander, AMCCOM, ATTN: AMSMC-QAR-R, Picatinny Arsenal, NJ 07806-5000. This description should include, but not be limited to, the procedures used, the accuracy and precision of the method, test data to demonstrate the accuracy and precision and drawings of any special equipment required (see MIL-I-45208).

6.4 Subject term (key word) listing.

Propellant
Antioxidant
Projectile

Custodian:
Army-AR

Preparing activity:
Army-AR

(Project 6810-A017)

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions – Reverse Side)

1. DOCUMENT NUMBER	2. DOCUMENT TITLE
MIL-M-70691	4,4' METRYLENEBIS

3a. NAME OF SUBMITTING ORGANIZATION	4. TYPE OF ORGANIZATION *(Mark one)*
	☐ VENDOR
b. ADDRESS *(Street, City, State, ZIP Code)*	☐ USER
	☐ MANUFACTURER
	☐ OTHER *(Specify)*: _____

5. PROBLEM AREAS

a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

6. REMARKS

7a. NAME OF SUBMITTER *(Last, First, MI)* – Optional	b. WORK TELEPHONE NUMBER *(Include Area Code)* – Optional
c. MAILING ADDRESS *(Street, City, State, ZIP Code)* – Optional	8. DATE OF SUBMISSION *(YYMMDD)*

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