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SUPERSEDING
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MILITARY SPECIFICATION

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

MIL-DTL-70691, dated 30 June 2020, is hereby reinstated and may be used for acquisition.

This specification is approved for use by the U.S. Army Combat Capabilities Development Command – Armaments Center (CCDC – AC), 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-diterbutylphenol) (see 6.1).

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3 and 4 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

Comments, suggestions, or questions on this document should be addressed to: Commander, U.S. Army CCDC – AC, ATTN: FCDD-ACE-QSA, Picatinny Arsenal, New Jersey 07806-5000 or e-mailed to usarmy.pica.ccdc-ac.list.ardec-stdzn-branch@mail.mil. Since contact information can change, you may want to verify the currency of this information using ASSIST Online database at https://assist.dla.mil.

AMSC N/A FSC 6810

DEPARTMENT OF DEFENSE STANDARDS

MIL-STD-286 - Propellants, Solid: Sampling, Examination and Testing
MIL-STD-1916 - DOD Preferred Methods for Acceptance of Product

(Copies of these documents are available online at https://quicksearch.dla.mil/.)

2.3 Non-Government standards and publications. The following document(s) form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents are those cited in the solicitation or contract.

ASTM INTERNATIONAL

ASTM E 300 - Standard Practice for Sampling Industrial Chemicals.

(Copies of these documents are available online at https://www.astm.org/.)

SAE INTERNATIONAL

ASM-STD-595 - Colors Used in Government Procurement

(Copies of these documents are available online at http://www.ams-std-595-color.com/.)

2.4 Order of precedence. Unless otherwise noted herein or in the contract, in the event of a conflict between the text of this document and the references cited, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Required inspections.

3.1.1 First article. When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.2.

3.1.2 Conformance. A sample shall be subject to conformance inspection in accordance with 4.3.

3.2 Chemical and physical requirements. The 4,4’-methylenebis (2,6-ditertbutylphenol) shall conform to the following requirements when tested in accordance with the applicable test method.

TABLE I. Chemical and physical requirements for 4,4'-methylenebis.

Property	Minimum	Maximum
Assay, mol %	98	---
Moisture, wt. %	---	0.5

3.3 Color. The 4,4'-methylenebis (2,6-ditertbutylphenol) shall be a white to light yellow powder between 37925 and 33793 of AMS-STD-595.

3.4 Workmanship. All materials shall be uniform in quality and free from lumps, grit, visible impurities, foreign matter, or other defects that would render the material unsuitable for intended use.

4. VERIFICATION

TABLE II. Requirements/verification cross reference matrix.

Method of verification 1 – Analysis 2 – Demonstration		3 – Examination 4 – Test				Classes of verification A – First article B – Conformance		
Section 3 Requirement	Description	Verification Methods				Verification Class		Section 4 Verification
		1	2	3	4	A	B	
3.1.1	First article	X		X	X	X		4.2
3.1.2	Conformance	X		X	X		X	4.3
3.2	Assay	X		X	X	X	X	4.4.1
3.2	Moisture	X		X	X	X	X	4.4.2
3.3	Color	X		X	X	X	X	4.4.3
3.4	Workmanship	X		X	X	X	X	4.5

4.1 Classification of inspections. The following inspection requirements specified herein are classified as follows:

a. First article inspection (see 4.2)
b. Quality conformance inspection (see 4.3)

4.2 First article inspection.

4.2.1 Submission. When specified (see 6.2), a sample shall be subjected to first article verification in accordance with Table III. Sampling procedures shall be conducted in accordance with 4.3.2 or 4.3.3. Samples shall be produced by the contractor using the same process procedures, material supply sources, production equipment, and production line as will be used in regular production.

Table III. First article acceptance tests.

Category	Examination or test	Requirement paragraph	Inspection method reference
Critical	None defined		
Major			
101	Assay, mol %	3.2	4.4.1
102	Moisture, wt. %	3.2	4.4.2
103	Color	3.3	4.4.3
Minor			
201	Workmanship	3.4	4.5

4.2.1 Rejection. If any sample fails to comply with any of the applicable requirements the first article quantity shall be rejected.

4.3 Conformance inspection.

4.3.1 Inspection lot formation. Inspection lots and batches shall comply with the MIL-STD-1916, paragraph "Formation and identification of lots or batches." A lot shall consist of one or more acceptable batches of 4,4'-methylenebis (2,6-ditertbutylphenol) produced by one manufacturer in accordance with one specification and revision under one continuous set of operating. Each batch shall consist of that quantity of 4,4'-methylenebis (2,6-ditertbutylphenol) that has been subjected to the same chemical and physical unit operations intended to make the final product homogeneous.

4.3.2 Sampling at the manufacturer. Samples shall be collected from the beginning, middle, and end of every batch in every lot in accordance with ASTM E 300. A composite sample of not less than 500 grams shall be formed which is assumed to be representative of that batch. All composite samples shall be verified in accordance with Table IV. Failure of a sample to comply with any of the requirements specified herein requires rejection of the batch represented by the sample.

4.3.3 Sampling at the receiving facility. Sampling shall be conducted in accordance with ASTM E 300. 4,4'-methylenebis (2,6-ditertbutylphenol) packaged in shipping containers (e.g. drums, boxes, etc.) shall be sampled using Verification Level I of MIL-STD-1916. Each container shall be sampled in two locations using a thief stick. The samples shall then be placed in a secondary container and mixed to form a composite sample consisting of a minimum of 500 grams for each lot. There shall be no reduced sampling. Every lot received shall be tested in accordance with Table IV. If any sample fails to meet any test requirement, the lot represented by the sample shall be rejected.

TABLE IV. Classification of characteristics.

4.3.4 4,4'-methylenebis (2,6-ditertbutylphenol).				
Category	Examination or test	Verification level	Requirement paragraph	Inspection method
Critical	None defined			
Major				
101	Assay, mol %	4.3.2	3.2	4.4.1
102	Moisture, wt. %	4.3.2	3.2	4.4.2
103	Color	4.3.2	3.3	4.4.3
Minor				
201	Workmanship	4.3.2	3.4	4.5

4.4 Test methods and procedures. Unless otherwise specified, all chemicals used shall be at least reagent grade.

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

4.4.1.1 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.

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

d. Oil bath, stainless steel cylinder, approximately 10-cm diameter and 10-cm deep. Can be made by cutting down a 1200-mL beaker.

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

f. Thermometer, total immersion, 150-160˚C, 10-25 cm total length, graduated to 0.1˚C with lowest graduation at least 9 cm above tip of thermometer.

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

4.4.1.2 Reagents and materials.

a. Nitrogen

b. Silicone Oil, 100 centistokes viscosity. Dow Corning fluids 200, 550, 710, or equivalent.

4.4.1.3 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 4.4.1.5. If a different type of thermometer is used, determine a stem correction for each set-point measurement. Apply the correction as directed in 4.4.1.5.

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 the set-point as described in 4.4.1.4. 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.

4.4.1.4 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 contaminants. 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. 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 the 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, 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 re-melted. 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: Re-melt 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.

4.4.1.5 Calculations.

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

$$\text{Corrected set-point, } ^{\circ}C = T_1 + C + B$$

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:

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

Where: A = height of mercury column of set-point thermometer, ˚C, see 4.4.1.3.
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.

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

4.4.3 Color. Verify by visual examination that the sample conforms with color requirement.

4.5 Workmanship. Take approximately 300 grams of the material and spread the composition out on a clean white surface. The sample shall be examined by personnel with normal vision or normal corrected vision. The sample shall be uniform in quality and free from lumps, grit, visible impurities, foreign matter or other defects that would render the material unsuitable for the intended use. Failure to meet the workmanship inspection or during following production processes shall result in batch rejection.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point’ packaging activities within the Military Service or Defense Agency, or within the military service’s system commands. Packaging data retrieval is available from the managing Military Department’s or Defense Agency’s automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. The 4,4'-methylenebis (2,6-ditertbutylphenol) is intended for use in M864 and XM1128 propellants.

6.2 Acquisition requirements. Acquisition documents should specify the following:

a. Title, number, and date of this specification.
b. Issue of ASSIST to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.1 and 2.2).
c. Provisions for submission/resubmission of first article samples.
d. Acceptance and description sheet - Acceptance and description sheets should be prepared for each lot in accordance with MIL-STD-1171.
e. Quantity required and delivery schedules.
f. Levels of preservation and packing (see 5.1).
g. Item hazard classification.

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 the equivalent test methods. A description of the proposed method should be submitted through the Contracting Officer to: Commander, CCDC AC, ATTN: FCDD-ACE-QEE, 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.

6.4 Subject term (key word) listing.
Propellant
Antioxidant
Projectile

6.5 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

Custodian:
Army – AR

Preparing activity:
Army – AR
(Project 6810-2020-015)

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at https://assist.dla.mil.