

INCH -POUND

MIL-DTL-43665D

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SUPERSEDING

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DETAILED SPECIFICATION

CLOTH, WOOL: MOTHPROOFING TREATMENT OF

This document is approved for use by all Department and Agencies of the Department of Defense (DoD).

1. SCOPE

1.1 Scope. This document covers the mothproofing treatment of wool stock, top, yarn or cloth.

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: Attn: DLA Troop Support, 700 Robbins Avenue, Philadelphia, PA 19111-5096. Since contact information can change, you may want to verify the currency of the address information using Acquisition Streamlining and Standardization Information System (ASSIST) online database https://assist.dla.mil/ .

AMSC N/A

FSC 8305

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DEPARTMENT OF DEFENSE STANDARDS

MIL-STD-655 – Provisions for Evaluating Quality of Cloth, Wool, Worsted and Wool Blends

(Copies of this document are available online at <https://quicksearch.dla.mil>.)

2.2.2 Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the solicitation or contract.

CODE OF FEDERAL REGULATIONS (CFR)

40 CFR 152 - Pesticide Registration and Classification Procedures

(Copies of this document are available online at <https://www.gpo.gov>.)

ENVIRONMENTAL PROTECTION AGENCY (EPA)

Federal Insecticide, Fungicide and Rodenticide (FIFRA)
(40 CFR part 162 State Registration of Pesticides Products)

Federal Insecticide, Fungicide and Rodenticide (FIFRA)
as amended by the Food Quality Protection Act of 1996 and the Pesticide Registration
Improvement Act of 2003

(Copies of these documents are available online at <https://www.epa.gov/pesticides>.)

FEDERAL TRADE COMMISSION

Rules and Regulations Under the Wool Products Labeling Act

Rules and Regulations Under the Textile Fiber Products Identification Act

(Copies of these documents are available online at <https://www.ftc.gov>.)

2.3 Non-Government publications. The following documents 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.

ASTM INTERNATIONAL

ASTM D1776/D1776M - Standard Practice for Conditioning and Testing Textiles
ASTM D3776/D3776M - Standard Test Methods for Mass Per Unit Area (Weight) of
Fabric

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

AMERICAN SOCIETY FOR QUALITY

ASQ/ANSI Z1.4 – Sampling Procedures and Tables for Inspection by Attributes

(Copies of this document are available online <https://www.asq.org>.)

INFORMA HEALTHCARE

Repeat Insult Patch Test-Modified Draize Procedure-
Principles and Methods of Toxicology, A. Wallace Hayes (editor)

(Copies of these documents are available online at <https://www.crcpress.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 herein, the text of this document takes precedence. Nothing in this document supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

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

3.2 Recycled, recovered or environmentally preferable or biobased materials. Recycled, recovered, or environmentally preferable, or biobased materials should be used to the maximum extent possible, provided that the material meets or exceeds the operational and maintenance requirements, and promotes economically advantageous life cycle costs.

3.3 Material.

3.3.1 Cloth document. The document governing the wool based cloth to be treated with the mothproofing compounds under this document shall be cited in the applicable acquisition document (see 6.2).

3.4 Mothproofing treatment. Mothproofing processing treatment for the cloth shall be an EPA registered product that complies with the Federal Insecticide, Fungicide and Rodenticide Act (FIFRA) (see 2.2.2). The mothproofing finish shall be applied uniformly to wool stock, top, yarn or cloth and strictly controlled to ensure that permethrin concentration level meets the requirements specified in Table I. The testing shall be as specified in 4.5. The treatment shall not degrade any performance characteristic of the cloth.

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TABLE I Mothproofing compound.

Mothproofing compound	Percent (%) weight of active ingredient of permethrin based on the weight of the wool fiber (%wt./wt.)	
	Min.	Max.
Permethrin (see 6.3)	0.0058	0.040

3.4.1 Mothproofing application. The wool content shall be mothproof treated in accordance with this document. If the total content of the white and colored wool is less than 15 percent of the total weight of the cloth, the wool may not need to be mothproofed. If the total content of white and colored wool is in excess of 15 percent of the total weight of the cloth, all the wool shall be treated with the mothproofing agent so that the percent by weight of the active ingredient permethrin is applied only the wool portion and meets the requirements specified in Table I when the cloth is finished.

3.4.2 Finished cloth. The mothproof finished cloth shall conform to the requirements of the applicable document for the untreated cloth. The percentage of active ingredient permethrin specified in Table I shall be based upon the weight of pure permethrin on the wool fiber content when tested as specified in 4.5.

3.5 Special marking.

3.5.1 Cloth and blanket. Each blanket or roll of cloth containing wool mothproof treatment in accordance with this document shall have the following information printed or stamped in black directly on the polyethylene bag across the center of the face or a white paper label inserted within the bag: "Mothproofed in accordance with MIL-DTL-43665". When piece labels/tickets are used, they shall include the word "mothproofed" in the nomenclature.

3.5.2 Fiber identification. Each roll of the finished cloth shall be labeled or ticketed for fiber content in accordance with the Rules and Regulations Under Wool Labeling Act and Rules and Regulations Under the Textile Fiber Products Identification Act.

3.6 Toxicity. The finished cloth shall not present a health hazard and shall show compatibility with prolonged, direct skin contact when tested as specified in 4.7. Chemicals recognized by the Environmental Protection Agency (EPA) as human carcinogens shall not be used.

3.7 Workmanship. The finished cloth shall be uniform in quality and shall conform to the quality of product established on this specification. The occurrence of defects as specified in 4.3 and 4.4, shall not exceed the quality acceptance levels as specified in the contract or purchase order.

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4. VERIFICATION

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

- a. First article inspection (see 4.2).
- b. Conformance inspection (see 4.3).

4.2 First article inspection. A first article, submitted in accordance with 3.1, shall be inspected, examined for appearance, color and finish defects in 4.4 and tested for the characteristics specified in 4.5.

4.3 Conformance inspection. Conformance inspection shall include the examination of 4.4 and the testing in 4.5 as applicable. Sampling for inspection shall be performed in accordance with ASQ/ANSI.Z1.4 and with acceptance quality limits as specified in the contract and/or order, except where otherwise indicated (see 6.2)

4.3.1 Inspection conditions. Unless otherwise specified, all inspections shall be performed in accordance with all the requirements of referenced documents, unless otherwise excluded, amended, modified or qualified in this specification or applicable procurement documents (see 6.2).

4.4 Marking examination. During the examination of the end item (cloth or blanket), each unit in the sample shall be examined for conformance to the special marking requirements (see 3.5). Each unit not marked as specified shall be a defect. The examination shall be in compliance with the "Wool Products Labeling Act" and the accompanying inspection and acceptance criteria specified in the untreated cloth or blanket end item document.

4.5 End item testing. In addition to the end item testing provisions of the untreated cloth document, the finished cloth or blanket shall be tested as required in Table II. The methods of testing specified, wherever applicable and as listed in Table II shall be followed. All test reports shall contain the individual values utilized in expressing the final test results. The sample unit and the sample size shall be as required in the end item testing provisions of the untreated cloth document. The lot shall be unacceptable if one or more sample units fail to meet any requirement specified.

TABLE II. End item tests.

Characteristic	Requirement Paragraph	Test Method
Identity of mothproofing compound	See 3.4	1/
Percentage of Active Ingredient Permethrin on Wool	Table I	See 4.6
Toxicity	See 3.6	See 4.7

1/ The contractor shall furnish a certificate of compliance to indicate which mothproofing compound was used and whether the treatment was applied to wool stock, top, yarn, or cloth.

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4.6 High Performance Liquid Chromatography (HPLC) testing procedure for (3-phenoxyphenyl) methyl (+/-)-cis, trans 3-(2, 2-dicholoethenyl)-2,2-dimethylcyclopropanecarboxylate (permethrin). The permethrin content of the mothproof treated wool cloth shall be evaluated in accordance with the following HPLC test method:

4.6.1 Active ingredient (pure) material content. This method determines the content of mothproofing agent as 100 percent (pure) permethrin material in milligrams per liter. This value is then mathematically converted to the content of active ingredient (pure) permethrin material as a percentage based on the weight of wool in the treated item (%wt./wt.). The permethrin standard shall be a $\geq 97\%$, mixture of cis/trans isomers. Permethrin standards are available from FMC Agricultural Products; Princeton, NJ 08543; FMC reference #33297; 97% purity/specified technical; AccuStandard; New Haven, CT 06513; reference # P-128N, or equal.

4.6.2 Test specimen. The specimen shall weigh 0.500 (± 0.200) grams and be conditioned as specified in ASTM D 1776/D1776M. Heavier weight specimen may be used if necessary. Adjustments will need to be made to solvent volume for extraction and calculations.

4.6.3 Number of determinations. Unless otherwise specified, two specimens shall be tested from each sample unit.

4.6.4 Apparatus.

- a. Pyrex culture tubes with closures
- b. Ultrasonic water bath
- c. 25 mL class A volumetric flasks
- d. 10 mL class A volumetric flasks
- e. High performance liquid chromatograph with UV detector, recorder, integrator, and reverse phase column. Manual injection valve or autosampler.
- f. Syringe for manual injection, if needed
- g. Analytical balance
- h. 10 mL class A volumetric pipette
- i. Disposable syringe
- j. Syringe filter (0.45 μm pore size)

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4.6.5 Reagents.

- a. Methanol, analytical or HPLC grade
- b. Water, HPLC grade
- c. 3-Phenoxybenzyl (1*RS*)-cis, trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane-carboxylate

4.6.6 Sample preparation and extraction procedure. The described extraction method removes 84 percent or more of the active material on the fiber. Aside from the active ingredient permethrin, the methanol extract can contain other extractable substances such as dyes. These have not been found to have any influence on the determination of the active content of permethrin. Other extraction methods, such as Accelerated Solvent Extraction (A.S.E.) or soxhlet extraction may be substituted.

1. Dry two (2) specimen per sample unit to a constant (± 0.001 grams) weight in a circulating air oven at a temperature of 221–230 °F. Cool in a desiccator prior to weighing.
2. Accurately weigh 0.500 (± 0.002) grams of textile material specimen in accordance with ASTM D3776/D3776M, Opt C. Record the actual mass of textile specimen.
3. Cut the specimen into small pieces and add to a respectively labeled culture tube.
4. Using volumetric pipette add 10mL of methanol to each culture tube and seal.
5. Immerse culture tubes in an ultrasonic bath for 90 minutes.
6. Cool to room temperature.
7. Using a disposable syringe and filter (0.45 μm pore size) place approximately 1 mL of the permethrin/methanol solution into a sample vial for analysis. Store remaining sample refrigerated at 39°F (4°C) for prolonged periods.

4.6.7 Preparation of standards.

The HPLC is calibrated using external standards prepared from an authenticated sample of pure active ingredient, permethrin. The molecular weight of permethrin is 391.28 g/mol.

1. Primary stock solution (400 mg/L permethrin in methanol).
 - a. Accurately weigh ~ 0.010 grams of authentic permethrin standard.
 - b. Record the actual mass of authentic standard.
 - c. Place into a 25 mL volumetric flask.
 - d. Dilute to 25 mL with methanol.
 - e. Calculate actual concentration in mg/L.

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2. Secondary stock solution (160 mg/L permethrin in methanol)
 - a. Pipet 4.00 mL of primary stock solution into a 10 mL volumetric flask.
 - b. Dilute to 10 mL.
 - c. Calculate actual concentration in mg/L.
3. Analytical Standard 1 (32 mg/L permethrin in methanol)
 - a. Pipet 2.00 mL of secondary stock solution into a 10 mL volumetric flask.
 - b. Dilute to 10 mL.
 - c. Calculate actual concentration in mg/L.
4. Analytical Standard 2 (24 mg/L permethrin in methanol)
 - a. Pipet 1.50 mL of secondary stock solution into a 10 mL volumetric flask.
 - b. Dilute to 10 mL.
 - c. Calculate actual concentration in mg/L.
5. Analytical Standard 3 (16 mg/L permethrin in methanol)
 - a. Pipet 1.00 mL of secondary stock solution into a 10 mL volumetric flask.
 - b. Dilute to 10 mL.
 - c. Calculate actual concentration in mg/L.
6. Analytical Standard 4 (8 mg/L permethrin in methanol)
 - a. Pipet 0.500 mL of secondary stock solution into a 10 mL volumetric flask.
 - b. Dilute to 10 mL.
 - c. Calculate actual concentration in mg/L.
7. Analytical Standard 5 (1.3 mg/L permethrin in methanol)
 - a. Pipet 0.080 mL of secondary stock solution into a 10 mL volumetric flask.
 - b. Dilute to 10 mL.
 - c. Calculate actual concentration in mg/L.

4.6.8 High performance liquid chromatography test. An Agilent 1200 series high performance liquid chromatograph is suitable for the determination. Other comparable equipment such as a gas chromatograph/mass spectrometer and column materials may be substituted. The suitability should be determined by making test runs with standard solutions of known concentrations.

4.6.8.1 Instrument settings. The following, or equivalent, parameters shall be used in analysis:

Column: Zorbax Eclipse XDB-C18 (4.6 x 150 mm, 5 μ m)

Eluent: 20:80 distilled water/methanol (Eluent should be filtered and degassed before use).

Flow rate: 2.2 mL/min.

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Column temperature: 104 °F (40 °C).

Injection volume: 20 µL.

Detector wavelength: 230 nm.

Analysis time: approximately 15 minutes.

Retention times: trans-permethrin, approximately 10.0 minutes;
cis-permethrin, approximately 12.8 minutes.

4.6.9 Calculations. The HPLC conditions employed here resolve the cis and trans permethrin isomers. Construct a calibration graph for each isomer using peak area. Determine a result for the two isomers present in each test sample by comparison with the calibration graph. Mean response factors calculated from the five standards should be used. The true result is a weighted average of the individual cis and trans results. This method of calculation allows the result for a multicomponent insect resist agent to contain contributions from all the active ingredients and compensates for any difference in the proportions of the two isomers between standards and test extracts.

The concentration of active ingredient permethrin in the test specimen is determined from the following relationship:

$$\text{Sample conc. mg/L} = \text{Sample PA}_t \times \text{SRF}_t \times \text{SWF}_t + \text{Sample PA}_c \times \text{SRF}_c \times \text{SWF}_c$$

Where:

PA_t = peak area of trans isomer

PA_c = peak area of cis isomer

RF_t = trans isomer response factor = $\text{conc.}/\text{PA}_t$

RF_c = cis isomer response factor = $\text{conc.}/\text{PA}_c$

SRF_t = standard mean trans isomer response factor

SRF_c = standard mean cis isomer response factor

WF_t = trans weighting factor = $\text{PA}_t/(\text{PA}_t + \text{PA}_c)$

WF_c = cis weighting factor = $\text{PA}_c/(\text{PA}_t + \text{PA}_c)$

SWF_t = standard mean trans isomer weighting factor

SWF_c = standard mean cis isomer weighting factor

The treatment level of active ingredient permethrin based on the percentage of wool in the treated item is determined using the following calculation.

$$\% \frac{\text{wt}_{\text{permethrin}}}{\text{wt}_{\text{wool}}} = \frac{V \times S \times \frac{1}{W} \times 100}{P_{\text{wool}}}$$

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Where:

V = volume of methanol in L

S = sample concentration in (mg/L)

W = original weight of the textile sample in mg

P_{wool} = percentage of wool in item

4.6.10 Report.

4.6.10.1 Active ingredient permethrin content. The percent active ingredient permethrin content of a sample unit shall be reported as the average of the values obtained for the two specimens tested and shall be reported with two significant figures. The individual values for each individual specimen used to calculate the average shall be reported to the nearest 0.0001 percent.

4.7 Toxicity test. Unless otherwise specified (see 6.2), an acute dermal irritation study and a skin sensitization study shall be conducted. When the results of the studies indicate the finished wool cloth is not a sensitizer or irritant, a Repeat Insult Patch Test shall be performed in accordance with the Modified Draize Procedure (see 2.3). If the toxicity requirement (see 3.6) can be demonstrated with historical use data on the finishing treatments used, toxicity testing may not be required (see 6.2).

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual 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 requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Department 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 mothproofing treatment of cloth is intended for use as a protective agent against moths in wool cloths, felts, and blankets used by military personnel of the Department of Defense.

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6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number, and date of this specification.
- b. The specific issue of individual documents referenced (see 2.2 and 2.3).
- c. When first article is required (see 3.1).
- d. When toxicity is required (see 3.6).
- e. Inspection conditions (see 4.3.1).
- f. Packaging requirements (see 5.1).

6.3 Mothproofing Agent. The permethrin is (3-phenoxyphenyl) methyl (+/-)-cis,trans 3-(2, 2-dicholoethenyl) -2,2-dimethylcyclopropanecarboxylatecis/trans ratio: min. 35% (\pm) Cis and max. 65 (\pm) trans. EPA registered products can be found under the trade name of Edolan ETS from StarChem LLC., Wellford SC; Perigen from Bayer Environmental Science, Research Triangle Park, NC and Evercide Industrial Mothproofer from McLaughlin Gormley King Company in Minneapolis, MN.

6.4 Subject term (key word) listing.

Mothproofed
Permethrin
Registered
Treatment

6.6 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 changes.

Custodians:
Army - GL
Navy - NU
Air Force - 11

Preparing Activity:
DLA-CT

Review activities:
Army - MD
Navy - MC

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