

INCH-POUND

CCC-D-950F

June 26, 1989

SUPERSEDING

Fed. Spec. CCC-D-950E

May 24, 1982

FEDERAL SPECIFICATION

DYEING AND AFTERTREATING PROCESSES

FOR COTTON CLOTHS

This specification is approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification governs dyeing and water-repellent and mildew-resistant aftertreating of cotton cloth.

1.2 Classification.

1.2.1 Types and classes. The finished cloth shall be of the following types and classes as specified (see 6.2):

Type:

- I - Vat dyed
- II - Mineral dyed
- III - Sulfur dyed (deleted see 6.7)
- IV - Vat printed
- V - Bleached
- VI - Resin bonded pigment printed

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: U.S. Army Natick Research, Development, and Engineering Center, Natick, MA 01760-5014 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 8305

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CCC-D-950F

Class:

- A - Water-repellent aftertreated
- B - Water-repellent and mildew-resistant aftertreated
- C - Untreated (except type II)
- D - Mildew-resistant aftertreated

2. APPLICABLE DOCUMENTS

2.1 Government documents. Unless otherwise specified, 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.

Federal Specification:

- PPP-P-1135 - Packaging of Duck Fabrics (Cotton; Synthetic Fiber; Cotton-Synthetic Fiber Blends)

Federal Standards:

- FED-STD-4 - Glossary of Fabric Imperfections
- FED-STD-191 - Textile Test Methods

(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards, and Handbooks as outlined under General Information in the Index of Federal Specifications and Standards and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, US Government Printing Office, Washington, DC 20402.)

(Single copies of this specification and other Federal specifications required by activities outside the Federal Government for bidding purposes are available without charge from Business Service Centers at the General Services Administration Regional Offices in Boston, New York, Washington DC, Atlanta, Chicago, Kansas City, Fort Worth, Denver, San Francisco, Los Angeles and Seattle.)

(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

Federal Regulations:Federal Trade Commission

## Rules and Regulations Under the Textile Fiber Products Identification Act

(The Code of Federal Regulations (CFR) and the Federal Register (FR) are for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-0001. When indicated, reprints of certain regulations may be obtained from the Federal agency responsible for issuance thereof.)

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

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless a specific issue is identified, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Association of Textile Chemists and Colorists (AATCC)

## Chromatic Transference Scale

(Application for copies should be addressed to the American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709.)

(Technical society and technical association documents are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

2.3 Order or precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document shall take precedence. Nothing in this document, however, shall supersede 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 (see 6.3) in accordance with 4.3.

3.2 Standard sample. The cloth shall match the standard sample for shade and appearance and shall be equal to, or better than, the standard sample with respect to all characteristics for which the standard sample is referenced (see 6.4).

CCC-D-950F

3.3 Materials. The specification governing the requirements for the untreated cloth to be dyed or treated under this specification shall be as cited in the applicable procurement document (see 6.2).

3.3.1 Cloth. The cloth shall conform to the requirements of the applicable specification for the untreated material prior to, and after dyeing and after-treating, except as otherwise indicated herein.

3.4 Physical requirements. Tests for the physical properties of the finished cloth specified herein shall be conducted as specified in 4.4.3.

3.4.1 Finished width. The minimum overall width of the finished cloth shall be as specified (see 6.2), with the following tolerances:

<u>Specified width of finished cloth</u>	<u>Tolerance</u>
Up to 45 inches	Plus 1/4 minus 1/4
46 inches to 58 inches	Plus 3/8 minus 1/4
59 inches to 66 inches	Plus 5/8 minus 3/8
Over 66 inches	Plus 3/4 minus 1/2

3.4.2 Yarns per inch.

3.4.2.1 Warp. The warp yarns per inch of the finished cloth shall be not less than 95 percent of the warp yarns per inch of the untreated cloth.

3.4.2.2 Filling. The filling yarns per inch of the finished cloth shall be not less than 90 percent of the filling yarns per inch of the untreated cloth.

3.4.3 Breaking strength. The breaking strength of the finished cloth shall be not less than 90 percent of the breaking strength of the untreated cloth in the warp direction and not less than 85 percent of the breaking strength of the untreated cloth in the filling direction.

3.4.4 Nonfibrous materials. All types except type V shall be suitably prepared prior to dyeing so that the dyed cloth shall contain not more than 2.5 percent starch and protein content including chloroform-soluble and water-soluble material. The type V cloth shall meet the same requirement after bleaching.

3.5 Type V, class C (bleached duck). The type V, class C cloth shall be bleached white to match the standard sample. The cloth shall be fully bleached and may be supplemented with optical brighteners which fluoresce to the blue region. The finished bleached cloth shall not discolor to a degree greater than that shown by the standard sample when tested as specified in 4.4.3. When a standard sample is not available, the discoloration shall be no worse than the rating of "fair" when tested as specified in 4.4.3.

3.5.1 Shrinkage. When preshrunk cloth is specified, the residual shrinkage shall be not more than 2.0 percent in either the warp direction or the filling direction when tested as specified in 4.4.3. The preshrinking process, if used, shall not be identified by name or trademark on the cloth, ticket, or package (see 6.2).

3.6 Color. The color of the finished cloth shall be as specified (see 6.2) and shall match the standard sample (see 6.4).

3.6.1 Matching. The color of the dyed or printed, treated or untreated, or the untreated bleached cloth shall match the standard sample when viewed under filtered tungsten lamps that approximate artificial daylight and that have a correlated color temperature of 7500 +/- 200 K, with illumination of 100 +/-20 foot candles, and shall be a good match to the standard sample under incandescent lamplight at 2300 +/- 200 K.

### 3.7 Dyeing.

3.7.1 Types I, IV, and VI. The types I and IV cloths shall be dyed with vat dyestuffs. Type VI cloth shall be printed using a resin-bonded pigment process. The use of dyes and compounds containing elementary sulfur capable of oxidation to sulfuric acid is prohibited. The dyestuffs shall be chosen and applied so that the dyed or printed cloth shall contain no more labile sulfur than that shown by the standard sample when tested as specified in 4.4.3 (see 6.5). When a standard sample is not available, the dyed and finished cloth shall show not more than a slight trace of labile sulfur as defined in the test method when tested as specified in 4.4.3.

3.7.1.1 Colorfastness types I, IV, and VI. The dyed or printed finished cloth, except for Sage Green 1509, shall show fastness to laundering (after 3 cycles), and accelerated weathering equal to or better than the standard or equal to or better than a rating of "good". Sage Green 1509 shall show "good" fastness to laundering (after 3 cycles), and "fair" fastness to accelerated weathering. The finished cloth shall show fastness to crocking equal to or better than the standard sample or shall have an AATCC Chromatic Transference Scale rating of not lower than 3.5. Testing shall be as specified in 4.4.3.

3.7.2 Type II. The cloth shall be dyed with a mixture of iron and chromium acetates. The use of compounds of the rare earth elements will be permitted provided that the colorfastness requirements are not adversely affected. The use of well-dispersed carbon black, vat dyes (sulfur free), and the phthalocyanine blue or green as required to produce a color which will match dark olive drab shades will be permitted. The finished cloth shall have a total ash content of not less than 3.5 percent when tested as specified in 4.4.3.

CCC-D-950F

3.7.2.1 Colorfastness type II. The dyed and finished cloth shall show fastness to laundering (after 3 cycles), and accelerated weathering equal to or better than the standard sample or equal to or better than a rating of "good". The finished cloth shall show fastness to crocking equal to or better than the standard sample or shall have an AATCC Chromatic Transference Scale rating of not lower than 1.5. Testing shall be as specified in 4.4.3.

### 3.8 Aftertreating.

3.8.1 Class A, water-repellent aftertreated. Cloth finished by class A process shall have an approved water-repellent finish (see 6.6). The water repellents shall consist of aluminum salts of saturated carboxylic acids (such as formate, acetate, palmitate or stearate), zirconium salts of such saturated carboxylic acids, or a combination of both, mixed with refined mineral and vegetable waxes, titanate esters, or a combination of both. The product shall be applied either in the form of an aqueous emulsion or in the form of a water free solvent solution.

3.8.1.1 Hydrostatic resistance. The hydrostatic resistance of the following listed finished cloths shall meet the requirements specified when tested as specified in 4.4.3.

	<u>Hydrostatic height (centimeters)</u>	
	<u>Average (minimum)</u>	<u>Single value (minimum)</u>
Army ducks, 10 oz. per sq. yd. and over	30	25
Army ducks, under 10 oz. per sq. yd.	25	20
Numbered ducks:		
No. 10 and lighter	30	25
No. 8 and heavier	35	30
Flat ducks	25	20
Twills	30	25

3.8.1.2 Spray rating (classes A and B, types I through VI). The measurements of the three individual determinations of the sample unit for spray rating for the classes A and B cloths shall be equal to or better than 80, 80, 70 when tested as specified in 4.4.3.

3.8.1.3 Colorfastness of class A cloths. The colorfastness of class A finished cloth shall be as specified in 3.7.1.1 and 3.7.2.1 for the applicable type.

### 3.8.2 Class B, water-repellent and mildew-resistant aftertreated.

3.8.2.1 Water repellent. Cloth finished by class B process shall be given an approved water repellent finish (see 6.6) to meet the requirements as specified in 3.8.1, 3.8.1.1 and 3.8.1.2.

3.8.2.2 Mildew resistant. Classes B and D finished cloths shall be given an approved mildew-resistant treatment (see 6.6) by the deposition of a mildew inhibitor in the cloth, as specified in table I and in the applicable end item specification or as specified in the procurement document (see 6.2). The minimum and maximum amounts of the inhibitor shall be as specified in table I when tested as specified in 4.4.3. Application of the mildew inhibitors shall be uniform and of good penetration. The color of the mildew compound applied shall not be supplemented by the addition of dyes and pigments.

TABLE I. Methods and materials for mildew resistant treatment 1/

Types	Inhibitor	Percent of inhibitor or copper on total weight of finished cloth	Method of application
I, IV, and VI	(a) 2, 2' methylene-bis-(4-chlorophenol)	1.10 min to 1.90 max	2-bath aqueous or solvent
I, II, IV, and VI	(d) Copper 8-quinolinolate in solubilized form	0.18 to 0.27 as copper	Solvent
I, II, IV, and VI	(e) Copper 8-quinolinolate in dispersion form	0.18 to 0.27 as copper	1-bath aqueous or solvent
I, IV, and VI	(h) A mixture of zinc salts of dimethyl dithiocarbamic acid and 2 mercaptobenzothiazole containing no less than 85 percent of zinc dimethyl dithiocarbamate	1.25 $\pm$ 0.25	2-bath aqueous or 1-bath aqueous dispersion

1/ The treatments listed are not interchangeable. End item specifications will specify which fungicide is to be used as well as the appropriate method of application and method of analysis.

CCC-D-950F

3.8.2.2.1 Mildew inhibitors. The Government reserves the right to specify, prohibit, or limit concentrations of inhibitors for requirements, such as toxicity, not specified herein. Unless otherwise specified, the cloth shall be mildew-resistant treated in accordance with the requirements shown in table I. Formulations containing mercury in any form are prohibited.

3.8.2.3 Colorfastness of class B cloth. The colorfastness of the dyed class B cloth prior to aftertreatment shall be as specified in 3.7.1.1 and 3.7.2.1 for the applicable type. The class B aftertreated finished cloth for all types shall have an AATCC Chromatic Transference Scale rating of not more than 1.5 when tested as specified in 4.4.3.

3.8.3 Class D, mildew-resistant aftertreated.

3.8.3.1 Class D cloth. The class D cloth shall be mildew-resistant treated in accordance with table I and shall meet the requirements as specified in 3.8.2.2 and 3.8.2.2.1.

3.8.3.2 Colorfastness of class D cloth. The colorfastness requirements for the class D cloth shall be as specified for class B cloth in 3.8.2.3.

3.8.4 pH. All class A and B treated cloths shall have a pH value not less than 5.5 nor more than 8.5 when tested as specified in 4.4.3.

3.9 Fiber identification. Each roll shall be labeled, and ticketed for fiber content in accordance with the Rules and Regulations Under the Textile Fiber Products Identification Act.

3.10 Marking. The face side of the cloth shall be identified by applying a stamping on that side of the cloth with the word "Face" at each end of the roll.

3.11 Length and put-up. The cloth shall be put-up in rolls as specified in 5.1. The minimum length of pieces shall be as specified in accordance with the untreated cloth specification.

3.12 Workmanship. The finished cloth shall conform to the quality established by this specification. The demerit points per 100 square yards when calculated as specified in section 4, shall not exceed the established maximum point value.

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 (examinations and tests) 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 this specification where such inspections are deemed necessary to ensure supplies and services conform to prescribed requirements.



4.1.1 Responsibility for compliance. All items shall 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 absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of ensuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling inspection, as part of manufacturing operations, is an acceptable practice to ascertain conformance to requirements, however, this does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to accept defective material.

4.1.2 Certificates of compliance. When certificates of compliance are submitted, the Government reserves the right to inspect such items to determine the validity of the certification.

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. When a first article is required (see 3.1 and 6.2), it shall be examined for the defects specified in 4.4.2 thru 4.4.2.3 and tested for the characteristics specified in table II.

4.4 Quality conformance inspection.

4.4.1 Component and material inspection. In accordance with 4.1, components and materials shall be inspected in accordance with all the requirements of referenced documents unless otherwise excluded, amended, modified, or qualified in this specification or applicable purchase document.

4.4.2 End item visual examination.

4.4.2.1 Yard-by-yard examination. Each roll in the sample shall be examined on the face side only. When the total yardage in the roll does not exceed 100 yards, the entire yardage in the roll shall be examined. When the total yardage in the roll exceeds 100 yards, only 100 yards shall be examined. All defects, as defined in section I of FED-STD-4, which are clearly noticeable at normal inspection distance (3 feet) shall be scored and assigned demerit points as listed in 4.4.2.1.1 with the following exceptions: Glossary numbers 1, 2, 3, 5, 14, 26, 27 and 29 shall not be scored unless they exceed three times the thickness of the yarn. Misdraws and reed marks shall be scored if they result in a clearly noticeable separation of warp yarns. For cloth lighter than No. 10 duck, only those knots and slubs which exceed the limits shown on figure 1 of FED-STD-4 shall be scored as defects. For No. 10 duck and heavier, a knot or slub shall be scored as a defect when it exceeds three times the normal yarn size. No linear yard (increments of 1 yard on the measuring device of the

CCC-D-950F

inspection machine) from any one roll within the sample shall be penalized more than four points. The sample size shall be 20 rolls, except, when the contract quantity is for 10,000 yards or less, the number of rolls shall be 8 for lot sizes of 3,200 yards or less and 13 for lot sizes of 3,201 yards up to and including 10,000 yards. The lot shall be unacceptable if the points per 100 square yards of the total yardage examined exceeds 40 points. The lot shall be unacceptable if the points per 100 square yards of two or more individual rolls exceeds 60 points. If one roll exceeds 60 points per 100 square yards, a second sample of 20 rolls shall be examined only for individual roll quality. The lot shall be unacceptable if one or more rolls in the second sample exceeds 60 points per 100 square yards. Point computation for lot quality and individual roll quality shall be as follows:

$$\frac{\text{Total points scored in sample} \times 3600}{\text{Contracted width of cloth (inches)} \times \text{Total yards inspected}} = \text{Points per 100 square yards}$$

(For type IV, when printed both sides, the alternate side shall be examined on every other roll in the sample.)

4.4.2.1.1 Demerit points. Demerit points shall be assigned as follows:

For defects 3 inches or less in any dimension	- one point
For defects exceeding 3 inches but not exceeding 6 inches in any dimension	- two points
For defects exceeding 6 inches but not exceeding 9 inches in any dimension	- three points
For defects exceeding 9 inches in any dimension	- four points

NOTE: The following defects when present, shall be scored four points for each yard in which they occur:

Baggy, ridgy, or wavy cloth  
 Color not uniform, off shade (not within established tolerance),  
 shaded end to end, side to side, side to center, or face to back  
 Patterns other than specified  
 Poor dye penetration, mottled, streaky, or cloudy  
 Nonuniform application of print colors  
 Overall application of compound not uniform  
 Objectionable odor  
 Clearly visible crystallization of mildew inhibitors  
 Not evenly bleached (type V)  
 Bias or bowed filling  
 Tackiness (sticky to touch)  
 Width not within specified tolerance

4.4.2.2 Length examination. During the yard-by-yard examination, each roll in the sample shall be examined for put-up and length in accordance with the provisions of the untreated cloth specification.

4.4.2.3 Roll identification examination. During the yard-by-yard examination each roll in the sample shall be examined for defects listed below. The lot shall be unacceptable if two or more of the following defects are present in the sample:

Preshrinkage process identified by name or trademark on the cloth  
or ticket  
Not labeled or ticketed in accordance with the Rules and Regulations  
Under the Textile Fiber Products Identification Act  
Face stamping missing from either or both ends  
Face stamping on wrong side

4.4.3 End item testing. The methods of testing specified in FED-STD-191, wherever applicable, and as listed in table II, shall be followed. The physical and chemical values specified in section 3 (except where otherwise indicated), apply to the results of determinations made on a sample unit for test purposes as specified in the applicable test method. The sample unit for testing shall be 1-1/2 yards of the untreated cloth, 1/2 yard of the bleached or dyed cloth prior to treatment and 3 linear yards of the finished cloth, all full width of the material. When shrinkage is required, the sample unit (finished cloth) shall be 5 linear yards. The sample size shall be as shown in table below except that for the inhibitor content, the sample unit shall be 1/4 yard and the sample size shall be five for all lots less than 22,001 units in size and eight for all lots over 22,000 units in size. The lot size shall be expressed in units of 1 yard. The lot shall be unacceptable if one or more sample units fail to meet any specified requirement, or when the lot average determination for change in yarns per inch or breaking strength does not meet specific requirements. When the data in the "Number of determinations per individual sample unit" and "Results reported as" columns are not specified in table II, they shall be as required by the referenced test method. All test reports shall contain the individual values utilized in expressing the final results.

<u>Lot size in yards</u>	<u>Sample size (sample units)</u>
800 or less	2
801 up to and including 22,000	3
22,001 and over	5

CCC-D-950F

TABLE II. End item tests

Characteristics	Require- ment reference	Test method	Number of determinations per individual sample unit	Results reported as
Yarns per inch:				
Warp	3.4.2.1	5050	---	1/ 1
Filling	3.4.2.2	5050	---	1/ 1
Breaking strength:				
Warp	3.4.3	5100	---	1/ 1
Filling	3.4.3	5100	---	1/ 1
Nonfibrous material	3.4.4	2611	---	---
Discoloration (type V)	3.5	5660 2/ 1	1	Pass or fail
Shrinkage in laundering	3.5.1	5550	---	---
Dye identification:				
Vat dye (types I and IV)	3.7.1	3/ 3	---	---
Resin bonded pigments	3.7.1	3/ 3	---	---
Presence of labile sulfur	3.7.1 and 3.7.2	2020	1	Pass or fail
Mineral dye (type II)	3.7.2	3/ 3	---	---
Ash content (type II)	3.7.2	2611	---	---
Colorfastness to:				
Crocking	4/ 4	5651	---	---
Accelerated weathering	4/ 4	5671 5/ 5	---	---
Laundering (after 3 cycles), (except types IV and VI)	4/ 4	5610 6/ 6	---	---

TABLE II. End item tests (cont'd)

Characteristics	Require- ment reference	Test method	Number of determinations per individual sample unit	Results reported as
Water resistance:				
Hydrostatic height	3.8.1.1	5514	---	---
Spray rating	3.8.1.2	5526	---	---
Presence of mercury	3.8.2.2.1	3/	---	---
Inhibitor content:				
Inhibitor (a) 2,2'- methylene-bis- (4-chlorophenol)	Table I	2011 7/	---	---
Inhibitor (d) and (e) copper-8-quinolinolate	Table I	2050 or 2060 7/	---	---
Inhibitor (h) mixture of zinc salts and 2 mercaptobenzothiazole	Table I	4.5.1	2	Average of 2 determinations to nearest 0.1 percent
pH	3.8.4	2811	---	---

1/ All test results shall be averaged, and this average value shall be utilized in calculating the percent change from the untreated cloth specification value. Results shall be calculated to the nearest 0.1 percent.

CCC-D-950F

TABLE II. End item tests (cont'd)

- 2/ Method 5660 shall be used, except that exposure of the specimen and standard sample shall be for 20 standard fading hours. Any specimen showing discoloration less than or equal to that of the standard sample shall be rated "Pass". Any specimen showing discoloration greater than that of the standard sample shall be rated "Fail". When no standard sample is available, the discoloration shall be considered satisfactory if it is not worse than the rating of "Fair" indicated in Method 5660.
- 3/ Unless otherwise specified, a certificate of compliance is required and will be acceptable for the requirement specified.
- 4/ See 3.7.1.1, 3.7.2.1, 3.8.1.3, 3.8.2.3 and 3.8.3.2.
- 5/ The time of exposure shall be 80 hours.
- 6/ The specimens must be dried after each of the 3 laundering cycles. Only the stain on the cotton fiber of the color transfer cloth shall be evaluated.
- 7/ The supplier shall certify that the specific inhibitor was used in the treatment of the cloth.

4.4.4 Packaging inspection. The inspection shall be in accordance with the quality assurance provisions of PPP-P-1135.

4.5 Methods of inspection.

4.5.1 Analysis of inhibitor (h) mixture of zinc salts and 2-mercapto-benzothiazole. The analysis of the treated material shall be obtained using either the colorimetric or electrolytic method as follows:

4.5.1.1 Colorimetric method.

4.5.1.1.1 Reagents and apparatus.

Soxhlet extraction apparatus, with thimbles.

Benzene (water white).

Concentrated ammonium hydroxide.

Copper sulfate solution, 2 percent.

Standard methyl cumate solution (copper salt of dimethyl dithiocarbamate).

4.5.1.1.2 Preparation of standard methyl cumate. Weigh 0.1 g of methyl cumate in a glass-stoppered weighing bottle. Transfer the cumate to a 1,000 mL volumetric flask rinsing with repeated portions of benzene. Dilute with benzene to 1,000 mL. Warm the solution, if necessary, and let stand overnight to insure completeness of solution. Prepare several Nessler tubes containing varying amounts of standard methyl cumate solution diluted to exactly 50 mL in a volumetric flask (e.g. 1, 5, 10, 15, 20, 25, and 50 mL of standard) with benzene. These Nessler tubes form a color range for comparison of the unknown.

4.5.1.1.3 Procedure. Cut the cloth sample into pieces about 1/4-inch square. Weigh 5 g of sample accurately to the nearest 0.001 g and place in an extraction thimble. Extract in the Soxhlet apparatus with 100 mL benzene for at least 4 hours or until the recycling solvent is no longer colored. Add 5 mL of concentrated ammonium hydroxide to the solvent, let stand for 15 minutes, and filter by suction through a Buchner funnel. Transfer the benzene-water mixture to a 250-mL separatory funnel, rinse the flask with benzene, add 10 mL of copper sulfate, and shake thoroughly. After separation is complete draw off the bottom water layer into a 150-mL beaker. Filter the top colored benzene layer by gravity into a 250-mL graduated flask. Extract the water layer again with about 50 mL of benzene and repeat the separation as before, collecting the colored benzene layer in the same flask. Continue the extraction until there is no color in the benzene layer. Add sufficient benzene to the flask until final volume is 250 mL. Transfer an aliquot portion into a Nessler tube and add a sufficient quantity of benzene to give a total volume of 50 mL. The aliquot shall be such that the resulting color shall fall within the range of the color standards. By comparison with the matching standard, determine the number of mL of standard equivalent to the diluted 25 mL of unknown. The absorbance of the final solutions of aliquots may also be measured on a suitable colorimeter at wave length of 440 millimicrons and compared to a previously prepared standard curve.

CCC-D-950F

4.5.1.1.4 Calculations.

$$\text{Percent inhibitor (h)} = \frac{\text{mL standard solution}}{\text{G} \times \text{D}} \times 2.55$$

Where:

G = Sample weight (g)  
D = Aliquot (mL) of colored benzene  
(prior to being diluted to 50 mL)  
from cloth sample

4.5.1.2 Electrolytic method.4.5.1.2.1 Reagents.

Concentrated sulfuric acid  
Concentrated hydrochloric acid  
Sodium hydroxide, 4N  
Ethyl alcohol, 95 percent  
Phenolphthalein indicator

4.5.1.2.2 Apparatus.

Electro-analyzer (see apparatus described in Method 2050 of FED-STD-191)  
(Injury to the platinum electrode can be prevented by first coating it  
with copper and then depositing the zinc on this surface.)  
180-mL electrolytic beaker  
Analytical balance  
Muffle furnace  
Bunsen burner  
Desiccator

4.5.1.2.3 Procedure. Weigh to the nearest 0.001 g a sample of approximately 5 g which has previously been cut into 1/4-inch squares. Place the sample in a porcelain crucible and ash in a muffle furnace at a temperature not exceeding 450°C (842°F). Cool and add 2 to 4 mL of hydrochloric acid and 4 to 6 mL of sulfuric acid. Evaporate until SO<sub>3</sub> fumes are liberated; cool, transfer to a 180-mL electrolytic beaker, and wash with distilled water. To this solution of zinc sulfate, add phenolphthalein indicator and sodium hydroxide solution until a permanent pink color is obtained. Then add 20 to 25 mL of 4N sodium hydroxide; dilute to 150 mL; filter if necessary.



4.5.1.2.4 Zinc content determination. Accurately weigh the cathode. Insert the cathode and anode in their respective holders, and place the beaker containing the zinc solution under the electrodes so that the cathode is almost completely immersed. Cover the beaker with a split watch-glass. Apply a current of 0.8 to 1.0 ampere and 3 to 4 volt electrode potential. After 3 hours the electrolysis is complete, provided that no more than 0.5 g of zinc was present. Without breaking the current, lower the beaker while placing a stream of water over the cathode to wash it free from electrolyte. Remove and dip the cathode in 95 percent alcohol. Flip to remove the excess alcohol. Dry in an oven at 105°C (221°F) for 5 minutes, cool in air, and weigh to the nearest 0.001 g. The increase in weight represents the weight of zinc in the sample taken.

4.5.1.2.5 Calculations.

$$\text{Percent inhibitor (h)} = \frac{\text{Wt. of zinc}}{\text{Wt. of sample}} \times 481$$

5. PACKAGING

5.1 Put-up and preservation. Put-up and preservation shall be level A or Commercial as specified (see 6.2).

5.1.1 Levels A and commercial. The cloth, shall be put-up and preserved in accordance with the applicable requirements of PPP-P-1135.

5.2 Packing. Packing shall be level A, B, or Commercial as specified (see 6.2).

5.2.1 Levels A, B, and Commercial. The cloth shall be packed in accordance with the applicable requirements of PPP-P-1135.

5.3 Marking. In addition to any special marking required by the contract or purchase order, shipments shall be marked in accordance with the applicable requirements of PPP-P-1135.

6. NOTES

6.1 Intended use. The aftertreating processes are intended for dyeing, bleaching, and applying water repellent and mildew resistant finishes to cloth.

6.2 Ordering data. Acquisition documents should specify the following:

- a. Title, number and date of this specification.
- b. Type and class required (see 1.2).
- c. When first article is required (see 3.1, 4.3, and 6.3).
- d. Untreated cloth specification (see 3.3).
- e. Width of cloth required (see 3.4.1).
- f. Preshrinking when required (see 3.5.1)
- g. Color required (see 3.6).

CCC-D-950F

- h. Mildew inhibitor required when applicable (see 3.8.2.2 and 3.8.2.2.1).
- i. Levels of preservation and packing (see 5.1 and 5.2).

6.3 First article. When a first article is required, it shall be inspected and approved under the appropriate provisions of FAR 52.209. The first article should be a preproduction sample. The contracting officer should specify the appropriate type of first article and the number of units to be furnished. The contracting officer should also include specific instructions in acquisition documents regarding arrangements for selection, inspection, and approval of the first article.

6.4 Sample. For access to samples, address the contracting activity issuing the invitation for bids.

6.5 Dye formulation. The following dye formulation has been used successfully in vat dyeing shade Olive Drab 7:

Vat Black 25, CI 69525  
Vat Green 3, CI 69500  
Vat Green 8, CI 71050

If necessary, to obtain the proper shade a minimum of either or both of the following may be added:

Vat Yellow of suitable fastness such as:  
Vat Yellow 2, CI 67300  
Vat Brown 3, CI 69015

6.6 Approved treatments. Approval of the water repellent and mildew resistant treatments is the responsibility of U.S. Army Natick Research, Development, and Engineering Center, Natick, MA 01760-5014, and is based on extensive tests including those for toxicity which are not set forth in this specification. Because of the time required to evaluate and approve new treatments (approximately 6 months) only those chemical treatments already approved will be considered acceptable for the related procurement. Information pertaining to approval of new treatments should be obtained from the U.S. Army Natick Research, Development, and Engineering Center. The list of approved treatments may be obtained from the procuring activity.

6.7 Supersession data. The type III sulfur dyed cloth has been deleted since it is no longer applicable to cotton cloths.

6.8 Subject term (key word) listing.

Finishes  
Mildew-resistant  
Water-repellent

CCC-D-950F

MILITARY INTERESTS:

Custodians

Army - GL  
Navy - NU

Review Activity

Army - MD  
Navy - MC  
DLA - CT

User Activities

Army - ME  
Navy - YD

CIVIL AGENCY COORDINATING ACTIVITIES:

GSA-FSS  
HHS  
JUS-FPI

PREPARING ACTIVITY:

Army - GL  
(Project 8305-0271)



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U.S. ARMY NATICK RESEARCH, DEVELOPMENT  
and ENGINEERING CENTER  
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