

ENGINEERING EXPERIMENT STATION

Georgia Institute of Technology

PROJECT INITIATION

Date January 31, 1961

PROJECT TITLE: Research on Textile Standard and Grade Tolerances

PROJECT NO: 1-549

PROJECT DIRECTOR: A. M. Herron

SPONSOR: Department of the Air Force, Wright Air Development Division

EFFECTIVE: February 1, 1961 ESTIMATED TO RUN UNTIL: June 30, 1961

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Summary Technical Report (Final)

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GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA 13, GEORGIA

March 8, 1961



Headquarters
Wright Air Development Division
Air Research and Development Command
U. S. Air Force
Wright-Patterson Air Force Base
Ohio

Attention: WWRCHF-2

Subject: Monthly Letter Report No. 1, Project No. A-549
"Development of Shade Standard and Balanced Shade
Tolerances," Contract No. AF 33(616)-7907
Covering the Period 1 February to 28 February 1961

Gentlemen:

A summary of work done and progress during the month of February, 1961 is given below:

1. Acquisition of Materials for Dyeing Trials

Action was initiated to obtain requisite yardage of combed cotton sateen, conforming to MIL-C-547D, prepared for dyeing. Inquiry of major fabric producers disclosed that the Type I (9 ounce) sateen is available only from Galey and Lord at \$1.01 per yard. The Type II (7 ounce) sateen, however, may be obtained from J. P. Stevens from current production under a government contract. Their price was quoted at \$0.85 per yard. One other producer, Reeves Brothers, gave a quotation of \$0.85 per yard but cited a minimum order requirement (2000 yards). Although their quotation stated that their material, a typical commercial product, conformed to MIL-C-557D, it did not specify the type, 7 or 9 ounce. The price, quoted at \$0.85 per yard led to the assumption that their material is also the Type II, 7 ounce material.

Permission has been requested to substitute the less costly Type II sateen for the Type I specified in subject contract. This request is also based on speculation that conformance of the fabric to military specification would be more nearly assured if obtained from a plant presently manufacturing on a government contract.

Cost data and samples of candidate dyestuffs are being accumulated for use in this work.

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March 8, 1961

2. Exploratory Dyeing Trials

Trials have been conducted to establish optimum dyeing procedures in the use of laboratory equipment. Initial trials have shown that the plan to use a Callaway experimental slasher for pigment padding of narrow fabrics is, apparently, practical. Modifications of the laboratory pad-steam unit to be used in this work have been found necessary. Work is in progress to: (1) provide means to inhibit collection of condensed steam which, if allowed to drop on the dried, pigmented fabric, may cause spotting; (2) provide a creel and take-up mechanism for the 150-yard lengths of fabric to be used in developing the projected tolerance standards; and (3) provide improved means of draining condensate from the steamer unit.

Exploratory dyeing trials, using an improvised laboratory jig, have been made to enable performance of reproducible dyeing contemplated in Phase A.

Future Plans

Immediate future plans include continuation of laboratory scale dyeing trials utilizing samples of fabric presently on hand to be followed by dyeings using fabric selected by the sponsor for the final shade standard.

Respectfully submitted,

Arthur M. Herron
Project Director

Approved:

W. C. Whitley, Chief
Chemical Sciences Division

Addressee: 3 copies

bcc: A. M. Herron
W. C. Whitley
Library (2)
Dr. J. L. Taylor

May 5, 1961



Headquarters
Wright Air Development Division
Air Research and Development Command
U. S. Air Force
Wright-Patterson Air Force Base
Ohio

Attention: WWRONF-2

Subject: Monthly Letter Report No. 2, Project No. A-549
"Development of Shade Standard and Balanced Shade
Tolerances," Contract No. AF 33(616)-7907
Covering the Period from April 1 to April 30, 1961

Gentlemen:

Work was somewhat hampered during April by equipment failure. Laboratory pigment padding application of vat dye formulations requires the use of our Callaway experimental slasher for drying. The driving motor became defective and an exceptional delay of approximately 3 weeks has been experienced in its repair. The repair is now virtually complete and dyeing trials will be resumed.

A sample of fabric of the standard shade has been obtained. Bleached, mercerized and prepared-for-dyeing cotton sateen conforming to the specifications of MIL-C-357D has been obtained. The quantity estimated to be required for laboratory dyeing trials using narrow widths (6- to 7-inch) has been slit and prepared for laboratory pad-steam dyeing trials.

Selected vat dyes have been obtained for planned dyeing trials. Alternative components have been considered and provision made for their acquisition.

The primary effort during the previous month has been devoted to (1) completion of equipment modifications and repair of laboratory equipment to approximate full scale commercial practice, (2) exploration of techniques for instrumental appraisal and projection of dye requirements for attainment of balanced shade tolerances.

Instrumental measurement of shade difference using the Hunter color difference meter (on a different fabric) showed promise of the feasibility of shade differential projection by graphic representation. Visual (composite optical reaction) correlation, however, led to the exploration of alternative, perhaps supplementary, instrumental measurement techniques. Indications are that shading component requirements may be more readily projected by a combination of spectrophotometric data and the rectangular coordinate system employed with the Hunter color difference meter.

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May 5, 1961

Future Program

Laboratory dyeing trials using selected vat dyes will be resumed shortly.

Instrumental measurement and projection of dye formulations for shading purposes will be continued.

Respectfully submitted,

Arthur M. Herron
Project Director

Approved:

Wyatt C. Whitley, Chief
Chemical Sciences Division

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA 13, GEORGIA

July 10, 1961



Headquarters
Wright Air Development Division
Air Research and Development Command
U.S. Air Force
Wright-Patterson Air Force Base
Ohio

Attention: ASRCE-2

Subject: Monthly Letter Report No. 3, Project No. A-549
"Development of Shade Standard and Balanced Shade
Tolerances," Contract No. AF 33(616)-7307
Covering the Period from June 1 to June 30, 1961

Gentlemen:

A discussion of the work done during this period and problems encountered in this program was contained in a letter dated June 22, 1961, "Shade Standards and Tolerances for Textile Material-Cotton Sateen-Blue 517, Project No. A-549." The following is a brief summary of the work done during this month:

Laboratory scale dyeings have been made using several different formulations in addition to those described in Progress Report No. 2. Instrumental characterization of these dyeings has been made using a Hunter D-25 color difference meter. Nominal numerical differences in values were obtained on many swatches which were markedly different by visual appraisal. This led to speculation that measures should be taken to increase the sensitivity of the instrument or that other techniques for shade discrimination might be necessary.

Personnel of Hunter Associates Laboratory, Inc. made modifications to the color difference meter which were expected to increase its sensitivity and make shade discrimination of dark shades more precise. Revised instrumental data using the modified instrument have been obtained. A recording spectrophotometer was obtained on a loan basis for investigation of other techniques for shade discrimination in the dark, navy blue range.

The data from these two sensing systems are now being evaluated. Preliminary indications are that neither the increased sensitivity of the reflectance meter nor the spectrophotometric data afford shade discrimination characteristics approximating those obtained by visual comparison.

The laboratory scale dyeings, from which swatches were forwarded with the June 22 letter, are expected to afford a range of shades for selection by ASD of the shade standard and tolerance limits. Following notification of the selections, it was planned that a mill scale dyeing be made to duplicate the

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July 18, 1961

shade standard. This notification has been received and work initiated to effect the mill scale dyeing.

Future Program

The mill scale dyeing will be made to essentially duplicate the shade selected by ASD from laboratory scale dyeing swatches.

Further laboratory scale dyeings will be made using pad-steam equipment. Both original white goods and navy blue dyed material will be used to develop a range of shade tolerances.

Instrumental data will be obtained on these dyeings and such weighting factors as may be found applicable will be employed to afford correlation of instrumental and visual shade discrimination characteristics.

Respectfully submitted,

Arthur M. Barron
Project Director

Approved: 

 Wyatt C. Whitley, Chief
Chemical Sciences Division



GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA

August 11, 1961

Headquarters
Aeronautical Systems Division
Air Research and Development Command
Wright-Patterson Air Force Base
Ohio

Attention: ASRCEE-2

Subject: Monthly Letter Report No. 4, Project No. A-549
"Development of Shade Standard and Balanced Shade
Tolerances," Contract No. AF 33(616)-7907
Covering the Period from July 1 to July 31, 1961

Gentlemen:

This letter report is submitted in lieu of the Draft
Summary Report originally intended at this time. A no-cost
extension on this contract was authorized due to equipment
malfunction and efforts have been redoubled to enable com-
pletion of the work and preparation of the Draft Summary
Technical Report now scheduled for 31 August.

A summary of work done during this period is as follows:

I. Dyeing of Cloth

A mill scale dyeing using a typical pad-steam range at
Lowell Bleachery was made. A sample of this dyed material
was forwarded along with proposed tolerance standards (pre-
pared by laboratory-scale pad-steam dyeing process) with a
letter dated July 19. On examination by ASD the mill-dyed
sample was found to be unsuitable in shade--too thin and
green--necessitating reprocessing to correct the shade.

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It appears that (1) translation of laboratory-scale dye formulation for use on a commercial pad-steam range is subject to unanticipated variables, (2) the variation in hue occasioned by finishing treatments have placed severe obstacles in our efforts to produce a satisfactory shade standard, and (3) these unanticipated circumstances render many of the laboratory dyeings, intended for use as tolerance standards, of uncertain utility.

Three different formulations were developed in laboratory pad-steam dyeings to produce an approximation of the standard swatch provided by ASD:

	<u>Per cent dye in Pad Liquor</u>		<u>Represented by Swatch No.</u>
1.	3.5 Vat Black 9 (CI 65230)		
	1.6 Vat Blue 16 (CI 71200)	Dble Paste	27, 29
	1.5 Vat Blue 20 (CI 59800)		
2.	3.0 Vat Black 13	Dble Paste	
	2.3 Vat Blue 16 (CI 71200)	Dble Paste	41
	1.89 Carbanthrene Brilliant Violet 4B		
3.	3.0 Vat Black 13	Dble Paste	
	1.5 Vat Blue 16	Dble Paste	43
	1.5 Vat Blue 20		

Dye formulations for laboratory dyeings were developed by empirical means. Visual appraisal of resultant dyeings was used to adjust formulations to arrive at an approximation of the desired shade. A tabulation of the experimental dye formulations, each used in pad-steam dye applications, is shown in Table I. Swatches of each of these dyeings, correspondingly numbered, are being forwarded. It may be noted that a certain amount of shade difference exists between the

TABLE I

PAD LIQUOR COMPOSITION - EXPLORATORY DYEINGS

	Per Cent Dye in Pad Liquor						
Dyeing Number	<u>Cl 65230</u>	<u>Cl 59800</u>	<u>Cl 71200</u>	<u>Cl 59850</u>	<u>Carbanthrene Violet 4B</u>	<u>Vat Black 13</u>	
30	3.5				3.0		
31	3.5		1.0		3.0		
32	3.0		1.0		2.5		
33	3.0		2.0		2.0		
34			2.25	3.5	2.5		
35			2.25	3.5	2.25		
36			2.25	3.25	2.25		
37			2.5		2.5	3.0	
38			2.5		2.25	3.25	
39			2.5		2.1	3.0	
40			2.6		2.0	3.1	
41			2.3		1.89	2.8	
42		1.5	1.5			3.5	
43		1.5	1.5			3.0	

TABLE II

COMPARISON OF VISUAL AND INSTRUMENTAL DATA ON HUNTER METER

Dyeing Number ASD Std.	Visual Appraisal	L	a	b	ΔL	Δa	Δb	ΔE
30	Lighter ⁺⁺⁺ , Red ⁺⁺ , Blue ⁺⁺⁺	17.4	5.9	-8.95	L 1.25	R 1.4	B 3.6	4.06
31	Lighter ⁺⁺⁺ , Blue ⁺⁺⁺⁺	17.5	6.35	-10.1	L 1.35	R 1.85	B 4.75	5.23
32	Lighter ⁺⁺⁺ , Blue ⁺⁺⁺	18.4	6.05	-10.85	L 2.25	R 1.55	B 5.5	6.14
33	Light ⁺⁺ , Red ⁺ , Blue ⁺⁺	17.15	5.5	-9.0	L 1.0	R 1.0	B 3.65	3.91
34	Blue ⁺ , Lighter ⁺	16.2	4.45	-7.5	L .05	G .05	B 2.15	2.14
35	Lighter ⁺⁺ , Blue ⁺⁺	17.35	4.7	-9.85	L 1.20	R 0.2	B 4.5	4.6
36	Lighter ⁺ , Blue ⁺⁺	15.85	3.95	-7.15	D .30	G .55	B 1.8	1.9
37	Lighter ⁺ , Redder ⁺	15.65	4.65	-6.1	D .50	R .15	B .75	0.91
38	Darker ⁺⁺ , Redder ⁺	15.15	4.45	-4.0	D 1.00	G 0.05	Y 1.35	2.83
39	Darker ⁺ , Redder ⁺	16.45	5.05	-5.2	D 0.55*	G 0.55*	B .10*	1.01
40	Darker ⁺⁺ , Redder ⁺ , Duller	15.85	5.15	-3.1	D 1.15*	G 0.75*	Y 2.00*	2.42
41	Redder ⁺⁺	15.95	2.55	-5.85	Data not related to ASD Std. but 517-2			
43	Greener ⁺⁺	16.40	1.40	-5.10	Data not related to ASD Std. but 517-2			

*Compared with ASD Std. values on day of observation: L 17.00 a 5.90 b -5.1.

Note: Above data taken with instrument not in constant temperature room, see Table III
data taken at constant temperature.

TABLE III

COMPARISON OF VISUAL AND INSTRUMENTAL TOPPED STANDARD

Dyeing Number	Visual Appraisal	L	a	b	ΔL	Δa	Δb	ΔE
517-2 Standard	(Proposed Standard Topped and Finished)	16.1	2.4	-4.2	--	--	--	--
29 F	Lighter ⁺⁺ , Bluer ⁺⁺	17.6	2.6	-7.0	L 1.5	R .20	B 2.8	3.19
38 W	Duller ⁺⁺ , Darker ⁺ , Redder ⁺	16.6	4.2	-2.7	L .50	R 1.8	Y 1.5	2.40
39 F	Bluer ⁺⁺ , Lighter ⁺⁺	16.5	2.8	-5.2	L .40	R .40	B 1.0	1.15
40 W	Redder ⁺⁺ , Darker ⁺	16.2	3.3	-4.8	L .10	R .90	B .6	1.09
41 W	Redder ⁺⁺ , Lighter ⁺	17.6	3.6	-7.0	L 1.5	R 1.2	B 2.8	3.4
43 W	Greener ⁺⁺ , Lighter ⁺⁺	16.4	1.4	-5.1	L .3	G 1.0	B .9	1.38

(1) In above table under the sample column, W indicates warp face and F indicates filling face.

(2) Instrument in constant temperature room.

TABLE NO. IV

COMPARISON OF INSTRUMENTAL DATA - FIRST PROPOSED STANDARD

Sample No.	Visual Appraisal	Reflectance Values - Hunter Meter						
		L	a	b	ΔL	Δa	Δb	ΔE
517-1		17.6	1.45	-5.45	--	--	--	--
29F	Thin Blue	17.35	3.3	-6.65	D 2.25	R 1.55	B 1.20	1.98
38W	Dull	15.2	1.6	-3.9	D 2.40	R .15	Y 1.55	2.86
40W	Full Red	15.15	2.0	-4.45	D 2.45	R .55	Y 1.00	2.70
41W	Thin Red	15.95	2.55	-5.85	D 2.65	R 1.10	B .40	2.90
43W	Full Green	17.05	1.0	-5.6	D .55	G 0.45	B .15	0.73

(1) In above table in the sample number column, W indicates warp face and F indicates filling face.

(2) Instrument not in conditioned atmosphere--results comparative on day of observation.

face and the back of the material. Efforts have been made to minimize this characteristic with only partial success. It is believed that the concentration of color on the face resulted from migration of the vat pigment emulsion during the drying operation, either because of nonuniform drying conditions or because of high wet-pickup of the emulsion. Increased squeeze roll pressures lessened but did not eliminate the defect. It is anticipated that the dyeings developed in laboratory dyeings for the shade tolerance standards be designated as face or back for visual and instrumental characterization in comparison with the shade standard.

Instrumental data taken with a Hunter color difference meter and visual comparison under natural (north) light is shown in Tables II, III and IV. The data contained in Table IV is included for information only.

The mill-scale dyeing which produced the unsuitable hue found in the sample forwarded on July 19 (hereinafter referred to as 517-1) was based on Formulation 3 above but with adjustment in the quantities of each of the dyes. "Strikes" were made on the full scale pad steam range to adjust the shade to that approximating Swatch No. 27. Four strikes were made followed by visual appraisal of the shade match. In the production run the formulation listed below was employed since the fourth strike produced a swatch approximating the secondary shade standard, Swatch No. 27, but slightly redder. The longer run through the machine, however, did not exhibit this

quality. Differences in short runs and longer ones are known to occur but the direction of change was, in this case, contrary to expectations based on the experience of the dyer and influence of formula variations in the several strikes on the machine. The final formulation used to produce 517-1 was:

Vat Black 13	3.59 ounces/gallon or 2.68 per cent of dye liquor
Vat Blue 16	3.46 ounces/gallon or 2.59 per cent of dye liquor
Vat Blue 20	3.13 ounces/gallon or 2.34 per cent of dye liquor

These differences may possibly be explained by the mechanisms of improved penetration and lessened migration of pad liquor during drying in the mill-scale dyeing.

When notice was received of the desirability for shade alteration in the proposed shade standard, 517-1, a quantity of the dyed material was returned to the finishing plant for topping with additional dye. It was impractical to reprocess the goods through the pad-steam range but, following mill custom for shade correction, the dyed material was padded with vat pigment, predominantly the Vat Blue 20, known to exhibit a reddening influence. The goods were then entered in a dye jig and treated with a reducing bath, caustic soda and sodium hydrosulfite. Swatches were removed after each two ends and dyestuff additions made in an effort to correct the shade.

Difficulty was encountered in that a large amount of the dye within the previously dyed goods was leached out and into the reducing bath in the jig. Additional pad liquor (1.00 ounce/gallon Vat Blue 20 and 0.50 ounce/gallon of Vat Black 13) was added to the leuco dye in the jig. When this failed to pro-

duce the original depth of shade in swatches removed from the cloth additions of Vat Blue 20 and Vat Black 13 were made. An aggregate of approximately 2 ounces per gallon of Vat Blue 20 and 0.36 ounce/gallon of Vat Black 13 were ultimately used in the jig topping bath. Swatches following oxidation and soaping appeared to be a virtual match to Swatch No. 27.

The cloth was then padded with a 4 per cent emulsion of Zelan AP, dried, and then cured at approximately 320°F, neutralized with an anionic detergent and soda ash. The washed and rinsed goods were taken while damp and compressively pre-shrunk.

The resultant material, now labelled 517-2, was altered in hue by the finishing processes so that in relation to either the ASD shade standard, a cotton twill, or to Swatch No. 27, an apparent greener hue is exhibited. A sample of this material is forwarded for consideration.

It is anticipated that further corrective action may require additional time, probably more than presently contemplated in the contract term. A draft of the Summary Technical Report is presently required by August 31, 1961, acquisition of additional fabric and dyeing and finishing may require 3 or more months. Indications are that the short yardage (with reference to typical mill pad-steam dyeing routine) may have to be dyed by the pad-jig method for economic reasons.

II. Instrumental Characterization of Shade Investigation

Reference Progress Report No. 2 and Letter dated June 22, subject "Shade Standards and Tolerances for Textile Material -

Cotton Sateen - Blue 517, Project No. A-549" investigation has been continuing. Since considerable difference in shade has been perceptible in colored materials whose instrumental color difference ΔE appeared to be within the limits considered ordinarily barely perceptible, efforts have been made to refine instrumentation techniques to improve shade discrimination.

Relocation of the color difference meter to a room which is maintained at a constant temperature has been found to eliminate certain discrepancies in instrumental readings taken on different days. Correlation of the L, a, and b units with CIE trichromatic coefficients obtained with a Bausch & Lomb recording spectrophotometer has not yet been accomplished. Expert advice in the use of the spectrophotometer for color difference measurement by the use of a reflectance attachment has been requested.

In an attempt to correlate the differences in color obtained with the Hunter meter with the coordinates of the MacAdam ellipse, a mathematical transformation has been made of the x, y planar coordinates to the rectilinear coordinates afforded by the Hunter reflectometer. The equations on which this transformation was made were derived using assumed values of the lightness, quality L, and x, y coordinates to locate the MacAdam ellipse representative of the area of the chromaticity diagram determined by conversion of L, a, and b data from the Hunter instrument. A plot of data derived from this transformation produced a flattened circle whose diameter was approximately 2 units in either the a or b axis. This appears to confirm

the previous assumption that 1.5 units in either direction should be an acceptable range of color difference when compared to a designated standard shade. Assumed values of L slightly at variance appeared to make little difference in the conformation of the transformation of the MacAdam ellipse to a and b coordinates. These data are somewhat lengthy and will be included in the Summary Technical Report on this investigation.

III. Future Plans

Future plans are somewhat contingent upon the decision as to acceptability of either the original mill-dyed goods 517-1 or the topped dyeing, 517-2, or whether additional goods and dyeings will be required.

Pending this decision, physical testing of 517-2 in continuing to ascertain its conformance to the specification requirements of MIL-C-557D.

Further dyeings and/or topping dyeings will be made to complete the shade tolerance range. Instrumental characterization of shade will be made and related to the ultimately selected shade standard.

Respectfully submitted,

Arthur M. Herron
Project Director

Approved: 

Wyatt C. Whitley, Chief
Chemical Sciences Division

PROGRESS REPORT NO. 1
PROJECT NO. A-549

RESEARCH ON TEXTILE STANDARD
AND
SHADE TOLERANCES

Arthur M. Herron

Engineering Experiment Station
Georgia Institute of Technology

February 1, 1961 - April 1, 1961

Printed April 18, 1961

Progress Report No. 1

Contract No. AF 33(616)-7907

A-549

Wright Air Development Division
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

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ABSTRACT

Work described in this report was exploratory leading to the development of dye formulations and laboratory techniques to simulate commercial practice in the development of a shade standard for a dyed cotton textile material. The ultimate objective is to develop dye formulations and application techniques for production of the shade standard and balanced shade tolerance standards with instrumental characterization thereof.

SECTION I

INTRODUCTION

Under Contract No. AF 33(616)-7907 work has been undertaken leading to the development of a shade standard and proportionately balanced shade tolerances for cloth, cotton, wind resistant, sateen, shade blue 517 (MIL-C-557D).

Specifications for the procurement of colored textile materials have traditionally contained requirements for shade and colorfastness equivalence to a standard provided by the purchaser. Such shade and fastness characteristics have sometimes been found difficult to reproduce in commercial dyeing practice. Inherent variations in the color characteristics of textile materials, coloring agents, coloring processes, and, basically, the color perception of the viewer, make quality control measures and determination of acceptable color variations somewhat arbitrary.

The objective of this investigation is to ascertain practical variations in dye formulations and processing variations to obtain coloration of the specified material within acceptable shade tolerances. Appraisal of acceptable variations devolves about definition of the tolerances and consideration of the measures necessary to obtain coloration by typical commercial practice. It is contemplated that candidate dye formulations will be developed by laboratory processes and characterized by instrumental means supplemented by visual comparison. Initial selection of dyes to be employed is being made on the basis of manufacturers' reports of fastness properties and cost considerations for the dyes and projected processing techniques. Instrumental appraisal of shade variations occasioned by dye or processing variations is being investigated.

During this initial period exploratory investigation has been made with regard to: (1) development of laboratory techniques and facilities suitable for appraisal of candidate dye formulations; (2) acquisition of suitable combed cotton sateen for laboratory, pilot-plant, and commercial scale dyeing; and (3) exploration, through mill and manufacturers reports, of candidate dye formulae and translation of laboratory formulations and application techniques to commercial practice.

Although material for the conduct of shade comparison of dye formulations on the specified fabric was not available during this period other material was used for instrumental comparison of selected variables. These variables, selected variations in dye bath concentrations in the dyeing of nylon, were compared by visual means and measurement using a Hunter color difference meter. Conclusions therefrom, while not directly related to dyed cotton material, are expected to be used in the projections of variations in the experimental dyeings leading to the development of shade tolerance standards in this work.

SECTION II

WORK PROGRAM AND DISCUSSION

Procurement action has been taken to obtain the combed cotton sateen fabric to be used in this investigation. Preliminary investigation leading to development of candidate dye formulations has been undertaken. Dyeing trials, using available cotton fabric, have been conducted to establish a routine for the pilot plant dyeings to be conducted in the laboratory.

A. Phase A

Inquiry of commercial dyeing and finishing plant personnel and dyestuff manufacturers personnel has been made relative to likely dye formulations and their experience in projecting laboratory dye application formulations to commercial, full scale, dyeing practice. A number of navy blue formulations (using vat dyes), which were reported to possess good fastness properties, were obtained. Numerous personnel contacted related considerable success in the correlation of laboratory pad-steam trials with plant production formulation but many "implied" reservations with laboratory applications using other techniques. For this reason the originally contemplated laboratory dyeing trials using short lengths of fabric were given only an exploratory trial in the conduct of dyeings using a reduced-vat method.

A simulated jig was fabricated for use in dyeing short (1- to 2- yard) lengths to obtain sample dyeings for instrumental appraisal of color difference occasioned by variations in formulation. Several different dyeings were made wherein the percentage of selected components was varied in the reduced (leuco) vat, with the remaining conditions held consistent. Wholly unsatisfactory dyeings were obtained: dyeings with higher concentration of dyestuff

produced less coloration than some with lower concentration; dyeings with a preponderance of one hue component produced similar coloration to those with a component expected to produce a somewhat different hue; and, reproduction of coloration was not obtained with the same formulation and processing. These dyeings, made on a lightweight combed cotton sateen available in this laboratory, when perceived to be worthless for the planned instrumental color difference appraisal, were discarded. Similar lack of success was obtained with pigment-padded, ironer-dried lengths of cotton fabric which were reduced and developed in the simulated dye-jig.

Longer lengths, approximating 25 yards of 6-inch-wide cotton sateen (3 ounces per square yard), were then pigment-padded and dried using a Callaway experimental slasher. By this technique it was anticipated that migration of the pigment during padding and drying would be minimized and that handling inequalities during reduction of the dye could be virtually eliminated. Initial drying of the pigmented material was accomplished using two electrical radiant-heat-drying chambers (at 200° F and 230° F, respectively), followed by four electrically heated drying cylinders. To minimize dye-liquor requirements for these exploratory trials a special pigment emulsion trough was fabricated and attached before the twin squeeze rolls of the experimental slasher. The trough, which has a capacity of approximately 3 quarts, is expected to be used in the development of the shade tolerance standards ultimately to be produced (probably with certain pigment substituents). This method of padding and drying appears to be a reasonable replication of commercial practice and appears practicable for narrow fabric in the weights thus far employed.

Dye-jig reduction techniques were investigated on 10-yard lengths of fabric padded and dried as described above on the experimental slasher.

Although relatively uniform dyeings were obtained, successive trials using similarly pigmented material produced dissimilar shades, possibly due to undetected differences in the reduction process.

Employment of a laboratory pad-steam unit for production of all samples for formula comparison was then elected. The initial trials disclosed that certain modifications of the laboratory pad-steam unit were necessary because: (1) condensation (on ceiling pipes) of steam emanating from the unit caused droplets hazardous to dyeings; (2) drainage provision on the unit was found to be inadequate when long intervals of steaming were employed; (3) a special creel is required to accommodate long lengths of fabric ultimately to be employed in this investigation, and; (4) a take-up mechanism may be required at the delivery end of the steamer to minimize possible variation in processing of the steamed-but-not-oxidized material.

An absorptive material in an improvised tent to inhibit condensate droppings has been obtained, drains for each receptacle in the steamer unit have been installed, and a creel for rolls of pigment-padded narrow goods has been devised. Work is continuing to provide a take-up mechanism for steamed and washed goods in a fashion expected to simulate commercial practice.

Instrumental appraisal of vat dyeings on cotton thus far obtained appeared unwarranted because of their obvious unsatisfactory nature. In lieu thereof, instrumental appraisal of variations in dyebath concentration in acid dyeings on nylon fabric in an academic research problem was pursued. These data though not directly pertinent to this investigation, afforded an opportunity to explore a technique for graphic portrayal of color-difference versus dyebath concentrations and color component variations. In trials using a Hunter color difference meter indications were obtained that projection of dyebath component

variations may be plotted by graphic representation. These data, accumulated on a limited scale, are expected to be used in subsequent dyeings to be made in this investigation.

B. Phase B

Work in Phase B of subject investigation has been delayed by failure to obtain the required material for simulated commercial dyeing trials and by the equipment modifications found necessary.

SECTION III

FUTURE PROGRAM

Immediate future plans include:

1. Completion of equipment modifications to permit production of simulated commercial scale dyeings on narrow fabric cut from widths of the specified combed cotton sateen to be used in preparation of the ultimate shade standard;
2. Exploratory dyeings on laboratory equipment modified to simulate commercial dyeing practice: pigment-padding of selected dye formulations on 20- to 30-yard lengths of 7-ounce/square yard cotton sateen, using the Callaway slasher; steaming of these formulations following reduction bath; variation in shading components in the reduction bath (prior to steaming) by inclusion in their leuco (reduced) state; screening colorfastness tests on samples produced;
3. Instrumental appraisal and graphic projection of results to project the candidate formulations for development of the shade standard and the shade tolerance standards ultimately to be produced;
4. Continuation of efforts to produce an equitable basis for comparison of dye formulations on an economic (basic and with regard to processing requirements) basis.

Respectfully submitted:

Approved:

Arthur M. Herron
Project Director

✓ Wyatt C. Whitley, Chief
Chemical Sciences Division

PROGRESS REPORT NO. 2
PROJECT NO. A-549



RESEARCH ON TEXTILE STANDARD
AND
SHADE TOLERANCES

Arthur M. Herron

Engineering Experiment Station
Georgia Institute of Technology

April 1, 1961 - June 1, 1961

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Progress Report No. 2

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Wright Air Development Division
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

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ABSTRACT

A number of exploratory dyeings have been made on a laboratory scale but simulating commercial dyeing practice. Shade characteristics of the dyed material have been appraised by sight and by instruments. In certain instances the visual appraisal revealed obvious shade differences which were not indicated by the instrument. Measures to afford improved shade discrimination by instruments have been investigated. Further investigation is being conducted using both reflectometer and spectrophotometer data.

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SECTION I

INTRODUCTION

The development of a shade standard and 10 balanced shade tolerance standards using cloth, cotton sateen, in shade Blue 517 (MIL-C-557D) has continued. The objective has been to define the shade and tolerances by instrumental, numerical values.

Conferences with commercial dyers have revealed that the best measure of correlation may be obtained between laboratory dyeings and typical commercial dyeings by the use of pad steam units in each process. The literature reveals that instrumental characterization of small color differences has been made by a variety of techniques, principally through the use of reflectance or transmittance data obtained by spectrophotometric or reflectometric equipment. Correlation of visual appraisal of differences (detected by trained observers) with instrumental characterization has been variously successful. Different investigators have derived several formulas for the specification of the several interacting components of color matching. A discussion of various techniques, formulas for specification of color (in numerical terms derived instrumentally), and various elements of the science of discrimination of shade differences is contained in Color in Business, Science, and Industry, Deane B. Judd, published by John Wiley & Sons, New York (Ref. 1). In brief, he relates that visual detection of color difference involves several factors, variously defined, but essentially involving the hue, value (degree of saturation or depth), and chroma (Munsell unit which may be described in terms of variation in grey content, possibly as purity). In addition to these factors the nature of the illuminant used for viewing, visually or instrumentally, the background

(including reflective absorption and psychological factors), the chemical composition of the colorant, and the reflective/refractive character of the colored material being viewed frequently influence the discrimination of small color differences in materials.

An attempt is being made in this investigation to produce an acceptable shade standard which may be obtained by each of three different dye formulations and to define by instruments the acceptable limits of shade deviation which may be obtained by different shading variations during the dyeing process. Commercial dyers, using the skill derived by experience, often employ a combination of dyestuffs to obtain the desired shade and, in the event of processing or chemical behavior (of the dye-fiber system) differences, to correct shading deficiencies. In certain cases variations in the several components may produce unwanted behavior of the colored material: possibly inferior colorfastness under conditions of usage or undue metamerism (show of greatly different coloration in different, but probable, illumination), or processing difficulties (to obtain replicate coloration by typical means).

The instrument selected for use in this investigation is a photoelectric color difference meter developed by Dr. R. S. Hunter, described in Ref. 2. The integrating electronic circuitry of this instrument is reported to enable color measurement and discrimination in numerical terms which may be translated to the several geometrical forms widely recognized as descriptive of the perceptual ability of the "normal observer" (Ref. 2). Certain numerical limits of visual perceptivity were defined by MacAdam, (Ref. 3). It was related by Judd, (Ref. 1, pp. 260-264), that the many known factors involved in geometrical characterization of a given shade difference by the Hunter reflectometer conform to the Euclidean theory involved in specification of a color solid, hence

noteworthy for color discrimination of materials (MacAdam, Ref. 3). In Ref. 3 it is related that visual perception of color differences by tristimulus values (derived from spectrophotometric data) is somewhat dependent upon the locus of the dominant hue (in the visible spectrum) in a geometric representation of absorption/reflection characteristics of the "color solid" representing visual/radiant energy in the electromagnetic spectrum. In certain areas of the visible spectrum perceptual differences appear to be greater than in others but ability to measure and ascribe these differences is attributed to various reflectometric or spectrophotometric measurement devices. Formulas have been provided for conversion from one system of measurement to another to permit correlation of different measurement systems (Refs. 1 and 2). From Ref. 2 it appears that the rectilinear coordinate system inherent in the electronic circuitry of the Hunter color difference meter enables discrimination of shade difference by appraisal of the numerical value of ΔE (the square root of the sum of squares of the difference in $\underline{L}$, $\underline{a}$, and $\underline{b}$, between the standard and the sample for comparison) with acceptability limited to a composite difference of approximately 1.5 units.

Exploratory dyeings were made using laboratory equipment to simulate commercial practice in piece-dyeing of cotton material with vat (fast-color) dyes. In order to derive a basis for formula variation to produce shade gradients, initial, arbitrarily selected, dye formulations were investigated in trial dyeings and instrumental characterization of color characteristics was made. When the initial instrumental characterizations with the color difference meter were observed to be lacking in color discrimination attributes (with the dark shade under investigation) it was decided that spectrophotometric characterization might be obtained and a comparison made with reflectometric data. Significant differences in visual appraisal of shade as compared to apparent

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small color differences (which appeared to be within tolerable limits) by instrumental means appears to warrant re-examination of data and exploration of methods to refine color differentiation techniques (particularly in the dark (deep) shade under investigation).

SECTION II

WORK PROGRAM AND DISCUSSION

Modifications of laboratory dyeing equipment to permit simulation of commercial dyeing practice were completed during this period. Laboratory scale dyeings were completed to appraise the influence of varying dye-liquor composition and other processing variables to establish a suitable procedure for lengthy laboratory scale dyeings.

Elements of the dyeing process were:

1. Preparation of the fabric for dyeing: singeing, scouring, bleaching, and mercerizing. The cotton sateen procured for this investigation was thus prepared by the producer.

2. Pigment padding of an emulsion of selected vat pigments. Exploratory formulations are listed in Table I. In addition to the vat dye pigment each pad-liquor bath contained a penetrating agent. Padding was done on dual squeeze rolls on a Callaway experimental slasher, wet pick-up estimated at 70 per cent.

3. Drying and cooling of pigmented cloth. Drying was accomplished by passing the cloth between a series of electric radiant heating elements traversed by a current of heated air. This technique was found necessary to minimize migration of pigment during drying. Cooling of the fabric was effected by allowing it to remain on a specially designed wind-up beam at the delivery end of the slasher.

4. Padding of pigmented goods with a reducing liquor containing caustic soda, sodium hydrosulfite, and retarding agents--dextrin and sodium nitrite. This padding treatment was accomplished on a specially designed laboratory pad-steam unit.

TABLE I

PAD LIQUOR COMPOSITION - EXPLORATORY DYEINGS

Dyeing Number	Per Cent Dye in Pad Liquor				Total
	C1 65230	C1 59800	C1 71200	C. Violet 4B	
5	1.00	1.67	1.67		4.33 ^{1/}
6	1.00		1.67	1.67	4.33 ^{1/}
7	1.33	2.33			4.33 ^{4/}
8	1.67	1.67	1.67		5.00 ^{1/}
9	1.67	1.33	1.33		4.33
10	1.67	1.67		1.33	4.67 ^{1/}
11	3.00	4.00			7.00 ^{1/}
12	3.00		4.00		7.00 ^{1/}
13	2.00	1.33	1.67		5.00 ^{1/}
14	1.67	1.33	2.00		5.00 ^{1/}
15	2.00	1.00	2.00		5.00 ^{1/}
16	2.00	2.00	1.00		5.00 ^{1/}
17	4.00		3.00		7.00 ^{1/}
18	4.00	3.00			7.00 ^{1/}
19	4.00	3.00	1.00		8.00 ^{1/}
20	4.00	1.00	3.00		8.00
21*	4.00		3.00		7.00 ^{1/}
22	3.50		2.75		6.25
23	3.50		2.75		6.25 ^{1/}
24	3.50	^{2/}	2.75		6.25 ^{2/}
25	3.00	2.00	2.00		7.00
26	3.00	2.00	2.00		7.00 ^{3/}
27	3.50	1.50	1.50		6.50
28	3.50	1.50	1.50		6.50 ^{1/}
29	3.50	1.50	1.50		6.50

* 200-yard dyeing -- partially stripped due undue depth.

^{1/} 13.3 per cent of pad liquor added to reducing bath.

^{2/} 0.8 per cent C1 59800 added to reducing bath.

^{3/} 1.0 per cent C1 71200 added to reducing bath.

^{4/} 0.67 per cent C1 59825 (Jade Green) added to pad liquor.

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5. Steaming in an air-free steam-heated chamber for approximately 20 seconds. The fabric passed through a cold water seal at the delivery end of the steam chamber which gave the first partial rinse.

6. Rinsing to remove excess alkali and unfixed dye particles.

7. Oxidation of the reduced vat (leuco) dye. Oxidation was accomplished in a laboratory dye-beck using an aqueous bath containing hydrogen peroxide and acetic acid.

8. Rinsing to remove oxidation solution.

9. Soaping to remove unbound dye particles and permit molecular realignment of dye in the fiber. A solution of anionic detergent and mild alkali, soda ash, was used in the dye-beck for a processing time of about 30 minutes.

10. Final rinsing and drying. Warm, then cold water rinses were employed in a dye-beck, followed by extraction and drying. Drying was accomplished by various means: on the slasher used for pigment padding, on steam-heated drying cans, in a tunnel dryer and in air.

It was found that the steaming conditions were somewhat critical to permit replication of dyeing results. After several unsatisfactory dyeings the optimum procedure was evolved. In all cases thus far, however, a certain amount of shade difference has been found between the face and the back of the cloth. Efforts are continuing to rectify this fault.

Swatches of the different dyeings were pressed smooth, cooled, then tested on a Hunter color difference meter. Numerical values of L, lightness (0 - black, 100 - white), a (redness - plus values, greenness - minus values), and b (blue - minus values, yellow - plus values) were obtained at several points in the fabric rotated at 90 degrees in successive readings. Average values were then obtained for the swatch provided by ASD and were compared with average values for the

several dyed swatches. The difference between each of the L, a and b values compared to the standard was computed. The composite shade difference was computed by the use of the following formula (Ref. 2):

$$\Delta E = \left[(L_s - L_x)^2 + (a_s - a_x)^2 + (b_s - b_x)^2 \right]^{1/2}$$

A tabulation of these values for dyeings 5 through 28 along with a visual appraisal of shade characteristics is contained in Table II. Dyeings 1 through 4 were experimental dyeings found unsuitable for appraisal.

Dyeings 5 through 16 were primarily intended to form a basis for formula variation to develop alternate basic formulas and variations to provide slight variation in resultant hue for shade tolerances. Instrumental characterization of dyeings 17 through 28 showed instrumental values originally expected to be within tolerable limits of variation but visual shade differences were obvious. These shade differences were in most cases greatly different from the target shade by visual examination. Examination under different lighting conditions also showed marked differences, some of which were believed to be objectionable.

In an effort to derive a basis for formula variation and/or dye component selection, investigation has been undertaken to obtain spectrophotometric data. Although the resultant hue is the quality observed by the Hunter color difference meter, it is reported that metameric qualities possibly contained in this hue are often undetected by this instrument. Preliminary observations of the spectral curves of selected dissimilar shades in the dark blue range under investigation disclose virtually overlapping traces. It is significant that to obtain spectral curves for the standard swatch as well as the experimental dyeings it has been necessary to use the high sensitivity range of the spectrophotometer

TABLE II
SHADE CHARACTERIZATIONS -
EXPERIMENTAL DYEINGS

Dyeing Number	Hunter Reflectance Values			Delta E	Visual Appraisal Difference (Depth and/or Hue)
	$L_s - L_x$	$a_s - a_x$	$b_s - b_x$		
5	- 0.0 -	R 1.35	B 3.75	3.99	Lighter - Blue
6	L 1.2	R 3.15	B 9.55	10.13	Lighter ⁺ - Redder, Blue
7	L 3.2	R 0.75	B 6.75	7.51	Lighter ⁺⁺⁺ - Blue
8	- 0 -	R 0.60	B 2.10	2.18	Same - Sl. Redder, Blue
9	L 0.7	R 0.20	B 3.20	3.28	Lighter - Greener, Blue
10	L 0.1	R 2.70	B 4.20	4.99	Lighter - Red=Purple, Blue
11	D 0.4	R 0.50	B 0.10	0.65	Darker - Redder
12	L 1.2	G 0.45	B 0.48	1.37	Lighter - Duller (no match)
13	L 3.3	G 0.15	B 3.75	5.00	Lighter ⁺⁺⁺ - Blue
14	L 2.7	R 0.55	B 3.65	4.57	Lighter ⁺⁺ - Blue
15	L 3.2	G 0.40	B 4.45	5.50	Lighter ⁺⁺ - Blue
16	L 1.95	R 0.80	B 3.53	4.10	Lighter ⁺ - Redder
17	D 0.65	G 1.70	Y 0.20	1.93	Darker - Duller
18	D 0.80	G 0.45	Y 0.70	1.15	Sl. Darker - Redder
19	- 0 -	R 0.30	B 0.10	0.32	Lighter - Redder (no match)
20	- 0 -	G 0.60	Y 0.50	0.78	Same - Redder, Dull (no match)
21	D 0.6	G 0.80	Y 0.80	1.27	Same - Greener (no match)
22	L 0.1	G 1.10	Y 0.30	1.12	Same - Greener (no match)
23	D 0.3	G 0.90	Y 0.60	1.11	Same - Greener (no match)
24	D 0.2	G 0.90	B 0.60	1.10	Same - Blue (no match)
25	D 0.25	R 1.10	B 1.15	1.62	Same - Sl. Redder, Blue
26	- 0 -	G 0.35	B 2.15	1.50	Same - Sl. Blue (near match)
27	D 0.4	G 0.30	B 0.60	0.78	Same - Match
28	L 0.75	G 1.10	B 1.40	1.53	Sl. Darker - Near match

Note: Letter prefixes indicate direction of difference - L, lighter; D, darker; G, greener (less red); R, redder; B, blue; Y, yellow (less blue) --- all with reference to the average values of the shade standard provided by ASD.

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(a Bosch and Lomb, Spectronic 505 with reflectance attachment), i.e., the 10 per cent transmittance setting. Typical settings to obtain tristimulus values (100 per cent setting) caused pen travel off the scale of the recorder chart. Efforts are continuing to ascertain methods for more meaningful instrumental shade discrimination, either by reflectometer (Hunter meter) or spectrophotometer.

SECTION III

FUTURE PROGRAM

Immediate future plans include

1. Continuation of piece-dyeing trials using different dye formulations to develop a range of shade variants.

2. Investigation of methods of instrumental analysis of shade characteristics of cloth dyed a dark blue approximating Blue 517. Techniques presently contemplated include provision of increased sensitivity with the Hunter reflectometer and variation of slit width and/or reference standard in use of a recording spectrophotometer.

Respectfully submitted:

Arthur M. Herron
Project Director

Approved:

Wyatt C. Whitley, Chief
Chemical Sciences Division

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SECTION IV

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TECHNICAL REPORT 61-



DEVELOPMENT OF SHADE STANDARD AND TOLERANCES
FOR CLOTH, COTTON, WIND-RESISTANT SATEEN, SHADE BLUE 1517

Arthur M. Herron

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

30 SEPTEMBER 1961

AERONAUTICAL SYSTEMS DIVISION

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ASD TECHNICAL REPORT 61-

DEVELOPMENT OF SHADE STANDARD AND TOLERANCES
FOR CLOTH, COTTON, WIND-RESISTANT SATEEN, SHADE BLUE 1517

Arthur M. Herron

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

SEPTEMBER 1961

Specialty Materials Section
Contract No. AF 33(616)-7907
Project No. 6314

AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

FOREWORD

This report describes work done by the Engineering Experiment Station of the Georgia Institute of Technology, 225 North Avenue, N. W., Atlanta 13, Georgia, under USAF Contract No. AF 33(616)-7907. This contract was initiated under Project No. 6314, "Research on Textile Standard and Shade Tolerances." The work was administered under the direction of the Materials Engineering Branch, Applications Laboratory, Directorate of Materials and Processes, Aeronautical Systems Division with Mr. W. L. Rooney acting as Project Engineer.

This report covers work conducted from February 1961 and completed in September 1961.

The dye formulations were developed by exploratory laboratory dyeings at the A. French Textile School, Georgia Institute of Technology. The shade tolerances were selected from the laboratory dyeings and related visually and instrumentally with the shade standard which was dyed at Lowell Bleachery, Griffin, Georgia. The invaluable assistance and cooperation of the staff of Lowell Bleachery, the Technical Laboratory of both E. I. du Pont de Nemours & Company and National Aniline Division, Allied Chemical & Dye Corporation is acknowledged.

The program was under the technical and administrative supervision of Dr. Wyatt C. Whitley, Chief, Chemical Sciences Division, Engineering Experiment Station, Georgia Institute of Technology. The Project Director has been Mr. Arthur M. Herron, Research Assistant. The editorial assistance of Mr. Frank Longshore, Technical Information Section, Engineering Experiment Station is acknowledged.

ABSTRACT

Shade standards and tolerances were developed for combed cotton sateen, MIL-C-557D, in shade Blue 1517. Alternative dye formulations were developed by laboratory dyeings made in a fashion to simulate commercial continuous dye practice. Visual and instrumental characterization of the standard and tolerances was accomplished. A multipurpose reflectometer was used for instrumental characterization of the dyed materials.

The shade standard was produced in a commercial finishing plant using a continuous dyeing system with minor shade correction using a batch method.

The tolerance spacing was determined by visual means and the shade difference as detected by the instrument was then related to the selected shade standard. Although a measure of correlation was obtained, a number of eccentricities were encountered. The report discusses some reasons for these deviations.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

W. P. Conardy
Chief, Materials Engineering Branch
Applications Laboratory
Directorate of Materials and Processes

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I. INTRODUCTION

Military clothing and textile materials are normally procured by the designated supply agency in accordance to specifications as to physical attributes, chemical requirements, and color characteristics developed by the Service which has the primary interest in the specific item.

Specifications for the procurement of colored textile materials have traditionally contained requirements for shade and color fastness characteristics equivalent to a standard sample provided by the purchaser. Such shade is generally chosen by selection of a basic shade and determination of optimum fastness characteristics available in textile colorants with consideration to commercial production practicality. It is often difficult to reproduce these characteristics in different plants due to differences in processing technique or to varied characteristics of colorants which may be used in them. In any variation in the color characteristics of textile materials, the coloring agents, dyeing processing conditions, and the basic color perception of the individual viewer make plant quality control measures and determination of acceptable shade variations somewhat arbitrary. The complex physics of color difference perception by the ultimate judge, the human eye, is further complicated by the chemistry of available colorants and the constitution of the material for coloring (dyeing). Various textile dyeing processes contain certain variables which may influence the resultant shade and, in apparently replicate dyeings, produce shade variations which may or may not be within tolerable limits.

The periodic replacement item purchases required to essentially duplicate items already in use by the military forces render the acceptable tolerances somewhat restrictive in nature. Thus the acceptability of small color differences in original materials is related to the color fastness character of even the color shading components.

Although color perception by the human eye must be accepted as the final authority, instrumental means of measurement have been developed which may supplant the judgment of the human eye and render the traditionally subjective comparison of small color differences more objective in comparative processes. Supplementary instrumental characterization of small color differences occasioned by slight, but known, variations in dye formulation or processing may thus prove to be valuable in both procurement inspection processes and in mill quality control measures.

In this investigation under Contract AF 33(616)-7907 it was anticipated that commercial dyeing practice and simulated commercial dyeing practice using a variety of dyestuffs and formulations, developed by empirical means, followed by instrumental characterization of shade differences would aid in the definition and/or prediction of acceptable variations in shade and performance characteristics.

The material specified by Aeronautical Systems Division for the standard sample, and thus the tolerances, was Cloth, Cotton, Wind-Resistant Sateen, Type II, Shade Blue 1517 (MIL-C-557D).

II. TECHNICAL BACKGROUND DISCUSSION

A. Dyeing of Cotton Textiles

Color in textile materials has long been the result of empirical formulations derived more from artistic-practical considerations than scientific development. Many advances have been made in recent years in the production of dyes possessing the desired colorfastness, ease of application, and uniformity of coloration but much dependence still devolves upon the efficiency and extent of quality control measures in the dyehouse.

Colorfastness considerations virtually limit selection of dyes for cotton fabrics for military apparel to vat dyes. The physico-chemical aspects of the dye-fiber system and the mechanics of vat dye application have been widely discussed in the literature. Commercial practice in cases of substantial yardage required of a given shade such as in a military procurement contract has tended toward the use of continuous dye ranges. These enable production of several thousand yards in a given processing lot, all of virtually the same shade. To accomplish this extent of standardization hundreds of yards may be in process before the results of the first several yards may be appraised.

Recent developments by laboratory equipment manufacturers have resulted in production of a laboratory steam-range to simulate the full-scale dye range. In this manner, then, laboratory dyeings have been reported to correlate with full scale range applications (with certain adjustments in formulation). Other vat dyeing techniques, involving the use of dye-jigs, which involve somewhat shorter yardage, have been reported with certain reservations as to reproducibility (from end-to-end and lot-to-lot shade characteristics) such that each lot of length amounting to 100 to 600 yards must be individually judged as to uniformity and shade matching character.

It has been related in the industry that a good measure of correlation may be obtained in translation of laboratory pad-steam dye formulations to full-scale production. Since each involve standardization of formulation in the padding operation and reproduction of the mechanical aspects of processing, it was elected that development of the standard and alternative formulations to produce the standard shade and set of tolerances be made using a laboratory pad-steam range. Arrangements were made for a mill-scale dyeing to be made on a continuous pad-steam range, customarily used to produce several thousand yards in a single lot, to produce a minimum of 200 yards of full width material to constitute the standard sample in the shade desired. It was found impractical to produce the shade tolerances on a mill-scale (pad-steam range) because of economic and practical considerations. Alternatively, it was found that the laboratory pad-steam range with modifications to permit processing of longer lengths of material would afford sufficient yardage for use as shade tolerance standards.

Numerous dye combinations are often used to obtain the desired shade but color fastness requirements generally limit the number of suitable combinations. If dyes known to produce good colorfastness properties are used, adjustments in formulation to attain the desired shade characteristics are often necessary.

Generally, in continuous dyeing practice, shade correction and development of the pigment pad formulations are accomplished by a series of "strikes." These are made by variation in the pad-liquor composition followed by processing to completion. A measure of shade correction is often accomplished by additions of vat dye pigment to the reducing bath, immediately preceding the steamer unit. The dye, reduced to its leuco form, may impart certain desired coloration, and it is generally found necessary to compensate for the loss of pigment from the padded and dried goods as they pass through the reducing liquor. An amount of the dye pigment is leached out of the goods in the reducing liquor, is reduced, and may influence the resultant shade, particularly near the start. An equilibrium condition between the added dye pigment in the reduction liquor and that which results from pigment leached from the impregnated goods generally results such that relatively uniform dyeings result in the processing of hundreds, even thousands of yards in a given lot.

The physico-chemical aspects of the dye fiber system have been widely discussed in the technical literature. It is generally conceded that the continuous (pad-steam, etc.) vat-dyeing process tends to minimize a number of the complex factors involved in the vat-dyeing process. Whereas time-temperature factors, the physical composition of the dyestuff and the fiber, the specific processing conditions, and many other varied influences may affect the time-honored vat-dyeing process on a dye jig, many of these are standardized in a pad-steam (continuous) process without particular regard to the predominant influence factor.

Within the dyeing and finishing industry it has been reported, through private communications, that a good measure of correlation may be obtained in translation of laboratory pad-steam dye formulations to full scale production. Correction of shade of materials dyed in the course of continuous dyeings is often made by a number of methods, stripping and redyeing, pad-jig vat dyeing (topping), wet- and dry-finishing techniques and others. Such corrections, generally involving only a few hundred yards, constituting the yardage required to establish the necessary equilibrium in the continuous process, are generally processed as small lots.

For the purpose of this investigation a requirement was that three different dye formulations be developed, each to approximate in shade and colorfastness properties a standard provided by Aeronautical Systems Division. In such developmental work a screening process using manufacturers' shade cards and basic hue characteristics of the component dyes was expected to involve a number of experimental dye applications followed by visual and instrumental appraisal of differences.

B. Instrumental Characterization of Shade

Developments in the field of optics and color science have led to the evolution of devices and techniques for visual and instrumental characterization of colors. Journals of the Optical Society of America contain many pertinent articles relative to color perception and color difference measurement, and the Munsell Book of Color (1929) affords a method for numerical characterization of a given color. Instrumental methods for color characterization include tri-stimulus spectrophotometry and rectilinear reflectometric measurement. In the

former, a system of three coordinates whose sum is unity (1) define the characteristics of a colored material in numerical units. This system of measurement, known as the CIE system, enables the definition of a specific color in planar coordinates, wherein knowledge of the two planar coordinates determines the magnitude of the third coordinate necessary to define a color in the requisite trilinear terms. Definition as such, however, may involve certain inaccuracies in visual perception of small color differences. Variations in instrumental characterization of visually equivalent colors (equivalent by consensus of several trained observers) were summarized by D. L. MacAdam,¹ wherein he concluded that color difference perception in terms of the acknowledged color solid concept involved extensive application of the principles of physical geometry. In his investigations it was shown that human perceptions of color differences were confined to certain limits in terms of tristimulus coordinates. Geometric projection of such limits about a selected color standard resulted in production of an ellipsoidal form, with the radii and axial dimensions varying greatly according to predominant color frequency wave length when plotted on a standard CIE chromaticity diagram and projected into the color solid assumed therewith. Specifications of small chromaticity differences with due respect to visual perceptivity limitations were suggested by MacAdam¹ wherein a circle of unit radius was inscribed on special oblique coordinate paper, the angular relationship of which was dictated by the formulas for derivation of the above-mentioned ellipsoids for the selected color standards. In this concept the minimum perceptible color difference was taken to be equivalent to one unit, the corresponding tristimulus coordinates of which varied in accordance with perceptual limits dependent upon the location of the primary color standard in the CIE chromaticity diagram.

Colorimetric discrimination through the use of optical filters was discussed in Circular 429, National Bureau of Standards, July 30, 1942, by R. S. Hunter.² The uniform color discrimination scales proposed therein led to the development of the multipurpose reflectometer, discussed in Journal of the Optical Society of America 48, 985-995 (1958).² Formulas are given for conversion of L, a, and b values obtained using the Hunterlab color difference meter to CIE tristimulus coordinates. Thus it appears feasible to instrumentally define color differences in dyed textile material. Since variation in chromaticity is specified to not exceed 1.5 MacAdam units, a range of tolerances could be prepared showing various acceptable colors at variance with the standard shade.

C. Eccentricities in Colorants and Colored Material

Colorants for textile materials frequently exhibit eccentricities as the concentration of dye in the fiber varies. Further, the basic hue of the colored material may produce a different reaction to the human observer as the background material or the light source is changed. For example, a blue dye may be comparatively blue in light shades but somewhat red as its concentration is increased. Another blue dye may exhibit a tendency to appear green as the concentration is increased. Metamerism, that characteristic of colored material to show differing characteristics under various conditions of viewing and illumination, is particularly acute in the case of dark blue, such as under investigation in this program. Thus visual sensitivity to shade differences, while ordinarily expected to correspond to instrumental measurement, was subject to additional influences

including psychological factors in the visual characterization. Employment of a complementary color for shading purposes may not necessarily produce the desired change but a dulling, a brightening, or change in hue not expected by logical deduction. For this reason instrumental measurement of small color difference may not reflect the magnitude nor the direction of the difference as observed by the human eye. It was anticipated that the determination of the magnitude of several components using the values of L , lightness; a , redness (plus) or greenness (minus), and; b , yellowness (plus) or blueness (minus), plus the projected color difference ΔE determined by the equation:

$$\Delta E = \left((L_s - L_x)^2 + (a_s - a_x)^2 + (b_s - b_x)^2 \right)^{1/2}$$

would define tolerable limits of shade difference in slightly (within tolerable limits for military clothing purposes) differently dyed or finished dyed cloth approximating the desired blue shade. The magnitude of minimum perceptible difference using these coordinates has been variously defined as between 1 and 4 units of ΔE .^{4/}

III. EXPERIMENTAL PROCEDURES IN THE DEVELOPMENT

It was anticipated that the mill-dyed shade standard would ultimately constitute the standard sample for some future military procurement actions, so a cotton sateen corresponding to the physical requirements of MIL-C-557D was obtained for dyeing trials from J. P. Stevens, Inc., which was then producing similar material in conjunction with another military procurement contract. This material, corresponding to the requirements of MIL-C-557D, "Cloth, Cotton, Wind-Resistant Sateen, Type II," was obtained after desizing, scouring, bleaching, and mercerizing according to their typical processing procedures except for the dyeing and water repellent finishing processes. Portions of the material were rolled and slit into 6-1/2-inch-wide rolls for dyeing trials on a laboratory pad-steam unit. The remainder was reserved for mill scale dyeing and finishing.

Laboratory equipment for the conduct of this investigation was modified as necessary and practicable to permit replication of typical commercial practice, but on a laboratory scale. Since usual employment of a laboratory pad-steam unit involves limited yardage, a special creel and improved doffing facilities were improvised on a laboratory pad-steam unit to permit processing of single lots 150 yards and longer. Pigment padding and drying was done using a modified Callaway experimental slasher with pad trough, dual squeeze rolls, two bands of blower-equipped electric radiant drying units and heated cans. A take-up mechanism was improvised to wind the cloth uniformly. Washing, oxidizing and soaping operations were performed on a laboratory dye-jig. Drying of the narrow material was performed on steam-heated drying cans.

Appraisal of shade of the various experimental dyeings was by both visual and instrumental means. A Hunter color difference meter, D-25, was used for measurement of differences in nearly matching successive dyeings.

IV. DEVELOPMENT OF DYE FORMULATIONS

The dye formulations used in this study were chosen for pad-steam application, to include a minimum of three different basic formulations to approximate a shade standard furnished by Aeronautical Systems Division. Each of the dyes selected were chosen, somewhat arbitrarily, on the basis of shading characteristics and manufacturer's report on colorfastness and ease of dyeing characteristics. Since the shading characteristics of different substituents in a basic pigment formulation may differ, a number of "strikes," dyeings on short yardage, were made, each followed by a standardized procedure for steaming, washing, soaping, etc. Thus the principal variable was the pigment composition in the pigment-padding operation. Visual and instrumental appraisal of the resultant shade was made and those formulations approaching the desired tolerances were used for dyeings on 150 to 180 yards of the narrow fabric. The formulation in laboratory dyeings which most nearly equaled the standard swatch was selected for the mill scale dyeing. Laboratory pigment formulations and the mill-scale formulation are shown in Table I.

Translation of the laboratory scale formulation to mill-scale processing required major modification in composition to obtain the requisite shade, which was found after full scale drying and preshrinking to be unsatisfactory. This material was then reprocessed by a pad-jig dyeing process with successive additions to the reducing liquor until the desired shade characteristics were obtained.

Water repellent finishing, however, again altered the shade but in a manner not anticipated, i.e., a greening. Since such shade alteration is not necessarily induced by some other water repellents (which might be used) it was believed that the shade tolerances developed in the large-scale laboratory dyeings would constitute a suitable range, when compared to the mill-dyed standard sample. A tabulation of the colorimetric characteristics of these dyeings related to the standard sample (that produced by jig-dye topping of the range-dyed sample found unsuitable) is shown in Table II. Dyeings obtained with different dyestuffs in exploratory trials to develop basic formulations showed wide disparity between instrumental and visual comparison. For this reason the spacing of the tolerances about the selected standard shade, with the several different formulations employed, was determined by visual means followed by instrumental characterization. This course appeared justified in view of the findings of Rizzo, et al, in fitting balanced tolerances to the MacAdam ellipse in other shade standard development work (WADD Technical Reports 60-151² and 60-294⁶). In this work a different instrumental measurement was employed but the ultimate judge was again the human eye. Practical considerations in the psychological reaction of the viewer when observing variations in shade under different illumination and with slight variations in background for viewing caused selection of dyed material for certain of the tolerances when comparison under other conditions would render it unsuitable. This circumstance may possibly be explained by the quality of dark blues to contain hue elements near both ends of the visible spectrum (and possibly influenced by the invisible portions at either end). An optimum balance of these qualities related to the shade standard resulted in the listing of visual and instrumental appraisal shown in Table II. Those dyeings which appeared to reasonably approximate the shade standard are described as to formulation and instrumental shade characteristics. In view of the multitude of water repellent finishes and their

varied influence on the dyed material only the mill-dyed material (in its full width) was subjected to this finishing treatment. The translation of the laboratory pad-steam dye formulations to mill scale operations is not claimed to be directly related, nor has any attempt been made to define the influence of subsequent finishing treatments should such translations be made.

V. COLORFASTNESS CHARACTERISTICS OF STANDARD SAMPLE AND SHADE TOLERANCES

Detailed colorfastness tests were conducted upon the mill-dyed and water repellent treated sample and are related in adjectival form in Table III. Since water repellent finishing treatment is recognized as influential on the laundering-, bleaching-, perspiration-, and crocking-fastness of vat dyed material, the shade tolerance samples were appraised for lightfastness only. Certain of the exploratory dyeings using each of the components involved in the shade tolerances finally developed were subjected to laundering, bleaching, and crocking tests in screening measures. In all cases the performance was good-to-excellent but time limitations prevented the completion of similar testing upon the shade tolerances ultimately selected.

VI. PHYSICAL PROPERTIES OF THE STANDARD SAMPLE

The physical properties of the standard sample (and representative of the shade tolerance samples) are recorded in Table III. Although detailed testing of the laboratory-dyed but commercially-manufactured shade tolerance samples was impractical it is believed that judicious choice of finishing materials and control over fabric handling would insure that such tolerance samples perform in at least equivalent fashion, substantially in conformance with the requirements of MIL-C-557D.

VII. DISCUSSION AND CONCLUSION

This investigation, principally performed on a laboratory scale but intended to simulate typical commercial continuous dyeing practice, revealed certain uncontrollable variables in the translation of formulations to the larger scale of operations. Variations in the dye formulation, techniques for application of dye-pigment, drying, steaming, washing, developing, washing, and other finishing treatments may lead to differences in resultant shade whether the fabric treatment is accomplished in mill-scale laboratory conditions or different mills.

Dye formulations employed in the development of the range of shade tolerances are shown in Table I. Instrumental and visual characterization of the standard shade and tolerances is shown in Table II.

VIII. LITERATURE REFERENCES

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IX. APPENDIX. TABLES

TABLE I

DYE-PIGMENT FORMULATIONS

Visual Character	Identification Number	Per Cent Dye in Pad Liquor (Color Index Number or Identification)				
		Vat Black 9 65230	Vat Blue 20 59800	Vat Blue 16 71200	Carb. Violet 4B	Vat Black 13 -----
		Laboratory Dyeings ^{1/}				
Full Blue	29	3.50	1.50	1.50		
Thin Blue	44			2.25	2.03	2.93
Full Green	43		1.50	1.50		3.00
Thin Green	45			2/	2/	2/
Full Red	40			2.60	2.00	3.10
Thin Red	41			2.30	1.89	2.80
Full Dull	38			2.50	2.25	3.25
Thin Dull or Full Std.	39			2.50	2.10	3.00
Thin Std.	46		3/	2/		2/
		Mill Dyeings ^{4/} (ounces/gal.)				
Thin Std.-2	1517-1		3.46	3.13		3.59
Standard	1517-2		5/	5/		5/

- ^{1/} Laboratory dyeings - Narrow fabric pigment padded on Callaway slasher, dried by electric radiant heaters, chemical pad (45 grams/liter caustic & hydro) and steamed in laboratory steam unit, 2 cold water washes, oxidized on jig using hydrogen peroxide and acetic acid, 2 hot water washes, soaping on jig using alkyl-aryl detergent.
- ^{2/} Topping dye process using Thin Standard-2 - Laboratory steam unit - dry dyed material run through chemical bath composed of (grams/liter): caustic and hydro, 45; Carbanthrene Violet 4B, 2.7; Cl 71200, 3.0; Vat Black 13, 3.9. After processing as in ^{1/}.
- ^{3/} Topping dye process using Thin Standard-2 - Laboratory steam unit - dry dyed material run through chemical bath composed of (grams/liter): caustic and hydro, 15; Cl 59800, 5; Cl 71200, 4; Vat Black 13, 5. After processing as above.
- ^{4/} Mill scale dyeing using pad steam range - material padded, dried in hot air, padded with chemical bath (240 gal. mix, 190 lb caustic, 95 lb hydro), steamed 20 seconds at 214°F, 2 cold water rinses, 2 oxidation boxes (180 gal, 40 lb acetic acid, 30 lb sodium bichromate, 2 lb anionic surfactant), 1 hot water wash, 1 hot water and anionic detergent wash, 1 hot water wash, dried on steam cans.

(Continued)

TABLE I (Continued)

DYE-PIGMENT FORMULATIONS

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- 5/ Topping dye process using Thin Standard-2 - pad-jig process in mill - pad liquor composed of 1.0 ounces/gallon Cl 59800 and 0.5 ounces/gallon Vat Black 13; additions to jig reduction liquor of 1.65 ounces/gallon Cl 59800 and 0.25 ounces/gallon Vat Black 13. 40 gallon reduction liquor containing 8 lb caustic and 6 lb hydro, with additions of total of 3 additional pounds of hydro as more dye was required for shading purposes. Total ends - 15. 2 ends cold water wash, 2 ends in oxidation bath (40 gallons containing 2 lb sodium bichromate), 2 ends hot water wash, 2 ends hot water wash, soaping in anionic surfactant (2 wash boxes 2 lb each), dried on steam cans. Water repellent finishing (4 per cent Zelan AP Paste buffered with sodium acetate) padded, dried, cured 2 minutes at 300°F, soaped (2 wash boxes 1 per cent anionic detergent and 1 per cent sodium perborate) 3 hot wash boxes, 1 cold wash. Compressively preshrunk on Sanforizer.
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TABLE II
INSTRUMENTAL CHARACTERIZATION OF SHADE

Visual Character	Identification Number	Instrumental Data						
		L	a	b	ΔL	Δa	Δb	ΔE
Standard	1517-2	15.80	4.02	-4.38	----	----	----	----
Full Std. or Thin Dull	39	16.28	4.75	-3.70	L 0.48	R 0.73	Y 0.68	1.16
Thin Std.	46	17.53	4.45	-4.88	L 1.73	R 0.43	B 0.40	1.83
Full Blue	29	16.28	5.48	-5.00	L 0.48	R 1.46	B 0.62	1.66
Thin Blue	44	19.08	6.00	-8.95	L 3.28	R 1.98	B 4.57	5.96
Full Green	43	17.63	3.75	-5.60	L 1.83	G 0.27	B 1.22	2.22
Thin Green	45	18.35	3.83	-6.53	L 2.55	G 0.19	B 2.15	3.34
Full Red	40	15.58	5.13	-3.93	D 0.22	R 1.11	Y 0.45	1.22
Thin Red	41	16.50	5.50	-5.63	L 0.70	R 1.48	B 1.25	2.06
Full Dull	38	15.87	4.68	-3.00	L 0.07	R 0.66	Y 1.38	1.53
Thin Dull or Full Std.	39	16.28	4.75	-3.70	L 0.48	R 0.73	Y 0.68	1.16
Thin Std.-2	1517-1	18.33	4.08	-5.45	L 2.53	R 0.06	B 1.07	2.75

Note: Values of L, a, and b were derived from average of 2 readings each in warp and filling direction of material. Prefix letters in ΔL , a, and b signify direction of difference: L - lighter, D - darker, R - redder, G - greener or less red, Y - yellower or less blue, B - bluer.

TABLE III

LABORATORY APPRAISAL OF SHADE STANDARD IN USAF BLUE 1517
CLOTH, COTTON, WIND-RESISTANT SATEEN
TYPE II, MIL-C-557D

<u>Characteristic</u>	<u>Test Method</u> (CCC-T-191b)	<u>Specification</u> minimum/max.	<u>Test Result</u>
Weave	Visual	5-H Sateen	5-H Sateen
Yarn ply - Warp	Visual	2-ply	2-ply
Filling		1	1
Yarns per inch - Warp	5050	120	134
Filling		88	91
Weight - ounces/yd ²	5041	7	7.13
Breaking strength - grab			
(pounds) - Warp	5100	130	137
Filling	5100	105	98
Tearing Strength -			
(grams - Elmendorf tester)			
Warp	5132	3000	2394
Filling	5132	3500	2707
Shrinkage in laundering - per cent			
Warp	5550	1	0.46
Filling	5550	1	1.16
Dynamic absorption - per cent			
Initial	5500	25	14
*Laundered	5500	35	30
Spray Rating	5526	90,90,80	100
Hydrostatic pressure - centimeters head			
Initial	5514	35	79
*Laundered	5514	27	59
Air permeability - cubic feet/minute/square foot			
Initial	5450	6.5	6.9
*Laundered	5450	6.5	6.9
Seam efficiency - per cent of breaking strength	5110	85	85-90
Colorfastness - Light (120 hours) excellent, Laundering-good, Bleaching-good, Perspiration-excellent, Crocking-good.			

* After laundering by Method 5610, modified to use double volume of wash water and 8 x 8-inch samples.