WADC TECHNICAL REPORT 54-611

IMPROVEMENT OF COLORFASTNESS PROPERTIES ON UNITED STATES AIR FORCE FABRICS

ROBERT J. PEIRENT

ROLAND E. DERBY, JR.

GEORGE O. LANGLAIS

LOWELL TECHNOLOGICAL INSTITUTE

RESEARCH FOUNDATION

FEBRUARY 1956

MATERIALS LABORATORY

CONTRACT No. AF 18(600)-182

PROJECT No. 7320

TASK No. 73202

WRIGHT AIR DEVELOPMENT CENTER

AIR RESEARCH AND DEVELOPMENT COMMAND

UNITED STATES AIR FORCE

WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Carpenter Litho & Prtg. Co., Springfield, O.
400 - 29 March 1956

FOREWORD

This report was prepared by the Lowell Technological Institute Research 'oundation under USAF Contract No. AF 18(600)-182. This contract was initiated ınder Project No. 7320, "Air Force Textile Materials", Task No. 73202, "AF :lothing Textile Materials", formerly RDO No. 612-13, "Textile Materials for Air 'orce Clothing", and was administered under the direction of the Materials ,aboratory, Directorate of Research, Wright Air Development Center, with Captain ł. D. Haire acting as project engineer.

The development of suitable dyeing formulae was accomplished by the Lowell Cechnological Institute Research Foundation. Specifications drawn from this levelopment were adhered to in dyeing the necessary stock to process the requisite ȷuantity of four (4) of the fabrics involved to fulfill the contractual commitments.

The dyeing and subsequent fabric manufacture were accomplished by:

'abric	Shade Number	Stock Dyer	Fabric Manufacturer
/ool, Serge	Blue 84	Barre Wool Combing Co.	Bachmann Uxbridge Worsted Co.
/ool, Gabardine	Blue 156	Barre Wool Combing Co.	Bachmann Uxbridge Worsted Co.
/ool, Gabardine	Gray 167	Elmvale Worsted Co.	H. B. Schwab Textile Corp.
/ool, Gabardine	Tan 193	Blackstone Dye Works	H. B. Schwab Textile Corp.

The scope of this study did not permit examination of all the dyestuffs produced lomestically or of all the possible dyestuff combinations which conceivably would ieet target properties; however, as many dyes and formulae as possible were included ıfter the usual screening methods were employed. During the course of this investiga-:ion, dyes and formulae may have been evaluated in conditions for which they were not .ntended. Hence, it must not be assumed that the results presented herein are equally 'alid for other test conditions or applications, nor is it to be construed that a dye ır formula is not entirely satisfactory for the manufacturer's intended use or ad-'ertised claim. Further, it is not to be construed that formulae other than those 'eported herein cannot perform equally satisfactorily. The disclosure of dye formulae, lyeing procedures, and methods of colorimetry does not constitute license for practice. 'he selection of a particular dye formula for producing fabric required in this project .oes not imply approval of the US Air Force for the specific dyes or formula for pro-.ucing the shades derived.

Grateful appreciation is extended to the following organizations for their ooperation and assistance in making possible an extensive investigation of dyeing ormulae:

Ciba Company, Inc.; General Dyestuff Corporation; E. I. du Pont de Nemours & Co., nc.; National Aniline Division, Allied Chemical and Dye Corporation; Sandoz Chemical orks, Inc.; and Textile Aniline and Chemical Company, Inc.

FOREWORD continued

The advice and suggestions of the following contributed greatly to the success of the work accomplished:

George Battye, Cyril J. Byron, Harry Clapham, John N. Dalton, Roland E. Derby, Sr., John Gould, Adolph I. Katz, Chester M. Kopatch, Hans Luttringhaus, Alex Morrison, Elmer Nelson, Leverett N. Putnam, Karl H. Schmatzler, Basil G. Skalkeas, Harold N. Webber, and Henry F. Widman.

This report covers the period of work from March 1952 to March 1955.

ABSTRACT

Dye formulae to provide improved colorfastness on the following United States Air Force fabrics were developed. When tested in a preliminary manner on laboratory materials, the formulae displayed the following colorfastness to light in comparison to that of the standards:

Cloth	Shade Number	Colorfastness of Developed Formulae Compared to Standards
Wool, Serge	Blue 84	Markedly superior
Wool, Gabardine	Blue 156	Markedly superior
Wool, Gabardine	Grey 167	Considerably better
Wool, Gabardine	Tan 193	Equal or slightly better
Nylon, Satin	Sage Green 511	Appreciably better
Nylon, Rayon	Blue 157	Equal or slightly better

The formulae developed for Blue 84 and Blue 156 withstand more than 80 hours Fadeometer exposure without producing a visual break in color.

The formula developed for Gray 167 displays only a moderate change in color after 140 hours of Fadeometer exposure --- an exposure that destroys the color of the standard almost completely.

Tests of the colorfastness of the 100-yard length of each of the first four fabrics listed which were produced using the developed formulae in the dyeing operations substantiated the laboratory-evaluated colorfastness results with one exception:

The color on the Tan 193 length was slightly less fast than the standard.

The last two fabrics listed, i.e., those involving shades Sage Green 511 and Blue 157, were carried through to the development of dye formulae --- no fabric was manufactured.

The development of the dye formulae, the problems encountered in establishing them properly in fabric manufacture, and the test measurements of the properties of the fabrics produced are presented.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

TABLE OF CONTENTS

LIST OF TABLES

LIST OF ILLUSTRATIONS

I INTRODUCTION

The investigation covered in this report was undertaken to develop dye formulations which would impart the best colorfastness properties to the following U. S. Air Force fabrics:

Shade	Fabric	Specification
Blue 84	Cloth, Wool, Serge, 15 oz.	MIL-C-849
Blue 156	Cloth, Wool, Gabardine	MIL-C-10176A
Gray 167	Cloth, Wool, Gabardine	MIL-C-6403
Tan 193	Cloth, Wool, Gabardine, Type IV	JAN-C-391
Sage Green 511	Cloth, Nylon, Satin	16211
Blue 157	Cloth, Nylon, Rayon	MIL-C-4072

The colorfastness properties of the formulae to be developed were to conform with the requirements of the appropriate government specifications and the terms of the subject contract. These requirements are summarized in Table 1. Of the requirements so specified, the colorfastness to light is the most rigorous and was the most difficult to attain. Because of this, considerable emphasis is placed upon colorfastness to light throughout the discussion which follows. This property is really the most important parameter of the investigation of dyeing formulations in this project.

Manufacture of one hundred yards of each of the above fabric styles (employing dye formulations to be developed) was required by the subject contract. Specifications for this phase of the contract are also given in Table 1.

Representatives of textile mills, the dyestuff industry and various research laboratories were invited to a meeting held at Lowell Technological Institute on June 3, 1952, for the purpose of discussing the project and obtaining a cross-section of the thinking of leaders in the textile industry concerning the goals set. At this meeting the advantages and disadvantages of various techniques and procedures were discussed at great length. Everyone proved interested and agreed to submit formulations which he thought were suitable in order that the Research Foundation might evaluate them. At the same time laboratory work was initiated within the Foundation to investigate available dyes in search of possible solutions to the problem. A brief discussion of the general procedure employed in this study is presented to indicate some of the difficulties inherent in the problem and to show the nature and solution of dyestuff formulation problems.

Since there are over 3000 dyes (associated with some 25,000 commercial names) from which a selection could be made, all existing data on the properties of dyes and the project requirements were carefully studied to avoid excessive laboratory experimentation. The more restrictions imposed on the results desired in each fabric, the smaller is the number of dyes applicable. Each condition added to the requirements places a greater restriction on the final choice of dyes and either makes the solution less confusing or impossible. Following is presented a fairly complete listing of considerations or conditions which must be examined when attacking a dyestuff formulation problem:

1. Fiber or fibers to be dyed.

2. Construction of the material to be dyed.

3. Stage of process at which dyeing is to take place.

4. End-use requirements.
   a. Lightfastness
   b. Wetfastness
   c. Crockfastness
   d. Perspiration fastness
   e. Fastness to gas fading
   f. Miscellaneous

5. The combination of dyes required to match the shade.

6. The cost of the formulation should be a minimum.

7. The formulation should be reasonably easily handled and capable of good control in the plant.

8. Type of dyeing machinery to be employed.

9. Severe metameric matches should be avoided.

10. Dyes should be available from several sources if large amounts are required.

It is apparent that each dyestuff has properties which make it either acceptable or not for each of the criteria. A study of the known properties of dyestuffs then serves to eliminate many of them. When the formulation is designed to replace an existing standard shade, such as in military fabrics, it is also very desirable to select a combination which produces a spectrophotometric curve which essentially matches that of the standard. This means the materials will match well under all lighting conditions. This, however, is not always possible. The solution of a dyestuff formulation problem attempts to give the best agreement between the desired properties and the specific properties of available coloring matters that will yield a desirable match. Under the present status of textile technology, a thoroughly trained colorist can, on the basis of his long experience, rapidly isolate a number of dyes in the area worthy of consideration. A limited number of experiments is then usually required to solve for the desired results within this area. In this project each shade was studied in accordance with these views.

In some cases the desired degree of colorfastness could not be obtained and a compromise in terms of lightfastness was necessitated. It is possible that the formulae selected do not represent the only possibility, and also that the discovery of new dyes will suggest new avenues for the development of other successful formulae. However, it is believed that the formulation developed for Shade Blue 84 and Shade Blue 156 represent as close to the theoretical optimum as ever obtained.

To permit ease of identification throughout the report, the various items developed and manufactured by the contractor and submitted to the U. S. Air Force are designated by the contractor's initials, such as "LTIRF formula" or "LTIRF fabric"; the fabrics selected by the U. S. Air Force as standards to be matched are designated in each case as "USAF standard" or simply "standard".

The numbers following the names of dyes appearing in the report are the Color Index number or the Prototype number taken, when listed, from The Technical Manual and Yearbook of the American Association of Textile Chemists and Colorists.

II DEVELOPMENT OF DYEING FORMULATIONS

The number of possible choices of wool dyes having the colorfastness properties was found to be quite limited. It should be noted that there is a large amount of published data on the properties of wool dyes and that these data were used advantageously to limit the scope of the laboratory experimentation. The possible dyes were then further screened on the basis of the two most rigorous requirements, namely, optimum lightfastness (both in the Fadeometer and in sunlight) and desired color match (spectrophotometrically). In this work considerable emphasis was placed on the spectrophotometric techniques of obtaining a match of the curve shapes of the dyes. In the case of those fabrics having a "mixy" appearance due to the blending of several different colored fibers (called primaries), each of the primaries was maintained at a color predicted to give a curve shape match to the blended fabric. Lightfastness of the individual fibers was used only as the first criterion for screening. Lightfastness tests of pads based on spectrophotometrically predicted matches were employed to attain the optimum lightfastness. When a desirable formulation was discovered, it was considered in view of the other requirements indicated previously. The spectrophotometric techniques which were employed in the work are treated in detail in the Appendix.

When testing for lightfastness, the Fadeometer was always calibrated by means of the Bureau of Standards "Standard Fading Paper" and, consequently, it is considered to approximate the standard fading hour within 5%. The approximate ratio of sun time to Fadeometer time was 1.5 to 1 for the same degree of change, viz., the exposure time was 60 hours in sunlight (between 9:00 a.m. and 3:00 p.m. on sunny days) and 40 hours in the Fadeometer.

A. Shade Blue 84, Wool Serge 15 oz.

This fabric requires blending of three or more different stock dye lots of fibers (one gray and two blue lots). Theoretically, one blue and one gray could be used but this would require such exact color control that this approach would be impractical. Three was selected as the optimum number of lots.

The initial work in the development of a formula for Shade Blue 84 involved the evaluation of fast-to-light blue and gray dyes which met the requirements as to availability, application method, wetfastness, and color requirements. These dyes were evaluated both in sunlight and in the Fadeometer. In all but one case the results in the Fadeometer paralleled those in sunlight, the exception being Algosol Blue O which is the leuco-ester of indigo. In this case the dye was a little less fast to the light of the Fadeometer than to that of the sun.

The dyes which were tested extensively are listed below. Those marked with an asterisk showed the best results.

For the blue shades:

Anthracene Navy Blue SWGG	CI 1059
*Acid Chrome Blue RRA	Pr 7
Mordant Blue B	Pr 93
Chromindigen Blue BRA Conc.	Pr 411
Anthracene Navy Blue SWR	CI 1062
Milling Blue BL	--
*Indigo or Helindon Blue B	CI 1177
*Algosol O (leuco-ester)	CI 1178

For the gray shade:

Alizarin Light Gray BBLW	Pr 206
*Solvat Gray BL	Pr 295
*Algosol Gray IBL-CF	Pr 295
Carbanthrene Gray GFL	

Shading colors:

Carbanthrene Violet BNX	CI 1163
Carbanthrene Blue GCD	CI 1112
Alizarin Violet N2R	--
*Alizarin Violet 3R	CI 1080
Alizarol Orange ML	Pr 457
Solvat Pink R	Pr 109
Solvat Orange R	CI 1217
*Anthraquinone Violet 3RA	CI 1080

When the colorfastness testing was completed, the properties of acid chrome Blue RRA and Indigo or its leuco-ester, Algosol Blue O, two blue dyes, suggested a very promising solution. Their very high fastness to light and their spectrophotometric characteristics indicated these dyes might fulfil all the requirements for the blue colored fibers in the fabric blend. This suggested a method for obtaining optimum fastness by techniques more simplified than those anticipated. These two dyes actually surpass all the requirements of the contract since they have excellent wetfastness properties and show only slight color breaks after 160 hours of Fadeometer exposure. In addition, the spectrophotometric measurements obtained during the preliminary work indicated (and later it was corroborated) that the USAF Shade Blue 84 could be matched by a simple three-primary combination: Two of these primaries to consist of separate dyeings, one with each of the blue dyes without any shading colors, the third primary (necessary to produce the "mixy" appearance) to be a gray shade. Solvat Gray BL was found to be superior in fastness properties to the other gray dyes and to be a suitable color for the third primary.

Laboratory dyeings and fiber blending established that by the proper selection of the concentrations of dyes these three primaries would give a color match of the standard in both daylight and artificial light. The blend was found to have outstanding colorfastness properties and to fade on-tone.

The two blue dyes are available from most major dye manufacturers. Both are low-priced and can be applied in conventional equipment. Acid Chrome Blue RRA can be applied easily, provided that iron, if present, is prevented from interfering with the chroming of the dye by means of a small addition of an organic sequestering agent to the dyeing solution. The purified forms of indigo can be applied with reasonable ease, although they do require a more exacting technique than do many of the chrome dyes. However, the same results as those obtained with Indigo can be obtained with less difficulty by using the leuco-ester form of indigo, i.e., Algosol Blue O. This can easily be applied. It is more expensive but not excessively costly

The gray dye Solvat Gray BL is available only from General Dyestuff Corporation and National Aniline Division of Allied Chemical and Dye Corporation. It is quite expensive (approximately $6.00 per pound). Since it is desirable to have all dyestuffs available from several sources and of reasonable cost, it was decided to transfer attention to the best chrome formula available. This consisted of Alizarin Gray BBLW and Anthraquinone Violet D. Although in direct comparison with Solvat Gray BL as a primary, this formula was less fast, and it was found that the three-primary fiber blends containing either gray were almost identical in lightfastness. Therefore, this chrome dye formula was finally selected as the gray primary.

The formulae finally evolved from laboratory experimentation and used for the production of the LTIRF fabric, described elsewhere in the report, are as follows:

Primary No. 1	- Blue	-	2.5% Indigo CI 1177
Primary No. 2	- Blue	-	2.1% Acid Chrome Blue RRA Pr 7
Primary No. 3	- Gray	-	0.315% Alizarine Light Gray BBLW Pr 206
			0.080% Anthraquinone Violet 3RA CI 1080

The blend required to produce a match to the standard is as follows:

33% Primary No. 1
35% Primary No. 2
32% Primary No. 3

The exact percentage contents for blending to match the standard may vary, depending on the shades of each primary; since slight differences are usually obtained in separate dyeings --- no doubt such differences will exist between the results of future applications of these formulae and those achieved in this contract --- some variation in the blending percentages or quantities of dyes employed on the primaries are possible and will necessarily be made to obtain a close match. The simplest procedure is, of course, to match the shades of the primaries closely during the dyeing and then modify the blending percentages slightly for the final shade.

Originally Indigo and Acid Chrome Blue RRA were selected because the combined shade of these changed the same as the standard in going from daylight to artificial light. However, the light change was found to be influenced by the gray fibers also. The proper light change is more easily obtained with Solvat Gray BL than with the chrome gray formula finally selected. This is due to the fact that the chrome formula does not become as red under artifial light as the Solvat. However, the use of the proper ratio of Alizarin Gray BBLW to Anthraquinone corrects this sufficiently so that the blend of primaries will produce a good daylight and artificial light match of the USAF Standard. The commercial application of this procedure required some time to solve but the laboratory formulations were converted successfully to mill formulae by the participating mill.

Another cheaper formula, a vat dye formula was discovered to be a good gray formula. This consisted of

Indigo	CI 1177
Indanthrene Brown RRD	Pr 121
Helindon Pink R	Pr 109

However, since this is only slightly better to light than the chrome dye formulation and exhibits considerably more difficulties in dyeing and shade matching, it did not merit further consideration.

Another blue chrome dye, Metomega Chrome Cyanine BLL or Alizarole Cyanone BLL, having good fastness to light (although not as good as Indigo), was identified. This could conceivably replace Indigo and would result in an all-chrome-dyed product. It would have greater fastness than the standard but not as good as the Indigo-Acid Chrome Blue RRA combination. At the time this phase was being undertaken, it was believed that dyestuffs not available from most domestic manufacturers should not be used; hence, this dye which did not meet this specification was not considered further. In addition, the goal was to achieve the maximum lightfastness practicably possible. The Indigo-Acid Chrome Blue RRA combination being appreciably better than any other formulation provided further justification for its selection.

All the dyes tested in the study of Shade Blue 84 and Shade Blue 156 are listed in Table 15.

B. Shade Blue 156, Wool Gabardine

The laboratory work on Shade Blue 156 paralleled that of Shade Blue 84 in that it was found possible to match the USAF Standard shade with the same dyes as those used for Shade Blue 84. These dyes had proved the fastest as described in the report of the development of Shade Blue 84. Although other combinations are definitely possible, the Indigo-Acid Chrome Blue RRA primaries best satisfied the criteria for Shade Blue 156. To match the standard for Shade Blue 156, the concentrations of Indigo and Acid Chrome Blue RRA are changed slightly from those used for Blue 84, the gray dyeing remains identical to that of Blue 84, but the amount of the gray fibers used in the blend is appreciably reduced. Following are the dyeing formulae and blending percentages required:

Laboratory Dyeing Formulae

Primary No. 1	-	Blue	-	2.0% Indigo CI 1177
Primary No. 2	-	Blue	-	2.0% acid Chrome Blue RRA Pr 7
Primary No. 3	-	Gray	-	0.315% Alizarine Light Gray BBLW Pr 206
				0.080% Anthraquinone Violet 3RA CI 1080

Blend

50% Primary No. 1
38% Primary No. 2
12% Primary No. 3

Both the laboratory and plant dyeing formulae are given in Table 3; the blend percentages are repeated in Table 4; and the dyeing procedure in Table 5.

All the dyes tested for lightfastness in the study of Shade Blue 84 and Shade Blue 156 are listed in Table 15.

C. Shade Gray 167, Wool Gabardine

The fabric to be produced in this project was to be stock-dyed. The nature of the fabric and its color are such that either piece- or stock-dyeing would be possible on normal production. It was, therefore, considered advisable to have available a formula suitable for both piece- and stock-dyeing but this qualification was not applied as a screening criterion until all formulations considered were evaluated.

Both Shade Gray 167 and Shade Tan 193 are of about the same depth of color and have the same fastness limitations because of the extreme lightness of their color. Thus, the investigations of these two shades were very similar.

A study of the fastness properties of dyes which satisfied the spectrophotometric requirements for Shade Gray 167 was made. The combination of dyes which were extensively investigated are as follows:

Combination 1

Alizarin Light Gray 2BLW	Pr 206
Alizarin Violet N2R	--
Acid Alizarine Red GWN	--
Milling Yellow 3G	CI 642

Combination 2

Solvat Gray BL	Pr 295
Solvat Pink R	Pr 109
Solvat Orange R	CI 1217

Combination 3

Algosol Gray IBL-CF	Pr 295
Algosol Brown IBR	Pr 118
Algosol Pink IR Fine	Pr 109

Combination 4

Carbanthrene Olive T Paste	Pr 547
Carbanthrene Brilliant Violet RK Paste	CI 1135
Carbanthrene Blue GCD Double	CI 1112

Combination 5

Alizarin Fast Gray 2BLW	Pr 206
Anthraquinone Violet 3RA	CI 1080

The last combination showed lightfastness equal to that of the others. Since it is a formula more generally acceptable for stock-dyeing and can also be used for piece-dyeing while the others are not, it was selected as the best formula. Although it did not quite meet the 80-hour exposure goal, it was appreciably better than the standard. Laboratory dyeings gave a 60-hour break and suffered only a moderate fade after 140 hours. This was considered excellent for such a light shade.

Experiments with the neutral-dyeing metallized dyes which have recently become available revealed no improvement of properties; hence, they were not pursued further.

The laboratory and plant dyeing formulae are given in Table 3; the dyeing procedure is described in Table 5. A complete list of dyes subjected to light tests is given in Table 16.

D. Shade Tan 193, Wool Gabardine Type IV

This fabric, for the purpose of this project, was to be stock-dyed. The nature of the color and fabric was such that either stock- or piece-dyeing is possible. It was considered desirable to have available for this shade a formula suitable for both piece- and stock-dyeing. However, this was not applied as a screening criterion until all formulations had been evaluated.

A study of the fastness properties of dyes which satisfied the spectrophotometric requirements for shade revealed that the lightfastness properties could not be made appreciably better than the USAF Standard for this shade. Slightly better lightfastness could be obtained with vat dyes but these lacked versatility and ease of application. The following formulation was selected as the best available. It was employed as a stock-dyeing formula but can also be used in piece-dyeing.

Alizarine Fast Grey BBLW	Pr 206
Anthraquinone Violet RA	CI 1080
Chrome Fast Orange 3RLA	--

The laboratory dyeings of this formula produced a break between 40 and 60 hours of Fadeometer exposure. This is equivalent or slightly better than the USAF Standard.

With such a light shade, it is extremely difficult to find dyes able to stand more than 40 Fadeometer hours.

The other combinations studied extensively follow:

Combination 1

Acid Alizarin Flavine R	Pr 1
Pontachrome Fast Orange L	--
Capracyl Gray GN	--

Combination 2

Carbanthrene Olive T Paste	Pr 547
Indanthrene Brown GAP Dbl. Paste	CI 1152
Indanthrene Red RK Dbl Paste	CI 1162

Combination 3

Alizarin Light Brown BL	--
Anthraquinone Blue SR	CI 1089
Brilliant Alizarin Milling Blue BL	--
Alizarol Orange ML	Pr 457

Combination 4

Algosol Brown IBR-CF	Pr 118
Algosol Gray IBL-CF	Pr 295
Algosol Golden Orange IRR-CF	CI 1098
Algosol Olive Green IB-CF	Pr 293

Combination 5

Alizarin Gray BBLW	Pr 206
Anthraquinone Violet D	CI 1080
Chrome Fast Orange 3RLA	--
Monochrome Red FGA-CF	Pr 300

It should be noted that although Combinations 2 and 4 displayed slightly better lightfastness, they were not suitable for piece-dyeing, and, being vat formulae, they are often considered undesirable for stock-dyeing. The increased fastness was not of sufficient consequence to warrant further attention.

A number of the new neutral-dyeing metallized dyes were tested; they did not provide any gain in fastness properties. They do decrease the dyeing time appreciably, but they are at present only suitable for stock-dyeing.

The laboratory dyeing formula and the plant formula employed in the manufacture of a 100-yard length of shade Tan 193 are recorded in Table 3; the dyeing procedure is described in Table 5. A list of all dyes tested for lightfastness is presented in Table 17.

E. Shade Sage Green 511, Nylon Satin

This material was to be piece-dyed. The initial phases of work involved the evaluation of all formulations submitted by dyestuff manufacturers and others concerned with this shade. Since it was felt that failure to meet lightfastness tests (i.e., exhibit fade equal to or better than the standard) would be an adequate screening test, all formulations were given 40 hours exposure in the Fadeometer. The following formulae were evaluated.

1.	0.04% Capracyl Yellow 3RD	--
	0.35% Capracyl Orange R	--
	0.50% Dupont Anthraquinone Green CN	CI 1078
2.	0.15% Acetamine Fast Yellow 4RL	--
	0.22% Celanthrene Fast Pink 3B	Pr 235
	0.04% Acetamine Scarlet B	Pr 244
	0.33% Celanthrene Brilliant Blue FFS Conc. 200%	
3.	1.80% Ponsol Olive GGL Paste	--
	1.80% Ponsol Direct Black 3G Double Paste	--
4.	0.40% Celliton Fast Blue FFRN Extra Conc.	--
	0.24% Celliton Fast Yellow 4RL Conc.	--
	0.64% Celliton Fast Pink RFD-CF	--
	0.50% Igepon T Powder 1 hour at 190°F	--
5.	0.50% Palatine Fast Blue BNDA-CF	Pr 318
	0.26% Palatine Fast Orange GENA-CF	Pr 315
	0.34% Palatine Fast Yellow GRNA-CF	Pr 316
	2.00% Formic Acid 1 hour at 200°F	
6.	0.44% Alizarin Fast Gray BLN New CF	Pr 206
	0.32% Acid Alizarin Brown RLL	--
	0.10% Supranol Yellow RA	CI 642
	1.00% Formic Acid 1 hour at 200°F	

		Per 100 gallons	
7.	Carbanthrene Grey GFL Paste	41.5#	--
	Carbanthrene Olive T Paste	36.5#	--
	Carbanthrene Brilliant Green Dbl. Paste	5.0#	CI 1101
	Pad-Jig method		

8.	0.150% Chromolan Blue NGG	Pr 144
	0.290% Alizarine Cyanone Green GN Extra	CI 1078
	0.750% Chromolan Orange GN	Pr 315
	0.125% Chromolan Red 3RB	--
	20.0% Glaubers Salt Crystals	
	5.0% Acetic Acid 28%	
	3.0% Formic Acid Conc. (to exhaust)	
	Dye 1 hour at 200°F	

Formulae Nos. 1 and 2 were judged to be the best, each being equal to or slightly better than the standard. Considerable difficulty was experienced in attempting to dye with these formulae. Very unlevel results were obtained in the laboratory dyeings. This was due to the nature of the fabric structure and the inability to treat the fabric in open-width form in laboratory apparatus. It is almost impossible to obtain completely level results on such a compact fabric composed of two styles of nylon yarn (70 denier Type 300 warp and 200 denier Type 100 filling). Fairly level results were obtained with a variation of Formula No. 2 when the dyeing time was extended. The following formula was selected:

0.34% Celliton Fast Blue FFRS Ex. Conc.	Pr 228
0.19% Celanthrene Fast Pink 3B	Pr 235
0.04% Celliton Scarlet B	Pr 244
0.14% Acetamine Fast Yellow 4RL	--
Boil 2-1/2 to 3 hours	

Open-width dyeing, such as on a jig, would be required with this fabric to obtain level results.

Formula No. 1 containing the new type of neutral-dyeing metallized dyes gave barre dyeings which could not be corrected, and, consequently, was eliminated even though its lightfastness was approximately equal to the acetate formula selected. Its color difference on fading 40 hours was 0.70 as compared to 0.64 for the acetate formula selected.

Evaluations of new dyes (as they became available) with respect to their applicability to this shade continued. A satisfactory dyeing was obtained using the following neutral dyeing metallized formula:

0.16% Cibalan Gray 2GL	--
0.09% Cibalan Yellow GRL	--

This formula gave even dyeings in a little more than half the time necessary for the acetate formula, its washfastness was better than that of the acetate formula, but its lightfastness was slightly less (40 hour C.D. = 0.82). The washfastness of the acetate dyeing was, however, equal to the standard. It was therefore concluded that either the acetate or the neutral dyeing metallized formula would be satisfactory.

There is, however, one serious drawback, namely, the difficulty or impossibility of finding more than one company making all the dyes in any of the combinations studied.

Considerable difficulty was experienced in finding a textile manufacturer supplied with or willing to make the yardage of fabric necessary to dye the one hundred yard length of fabric required. Because of this the plant dyeing phase of this shade was cancelled.

F. Shade Blue 157, Nylon Rayon

This material requires a union-piece dyeing formula which will produce exactly the same color on both the nylon warp yarn and the viscose filling yarn. This subjects the formula to a number of restrictions which considerably limit the number of dyes possible regardless of the level of fastness desired. There are available only a limited number of viscose dyes and nylon dyes which are compatible and permit dyeing procedures that are compatible; of those fulfilling these requirements only a few will give the same shade on both fibers. Unfortunately none of the dye combinations found to be technically satisfactory possessed both high lightfastness and excellent washfastness.

Following are listed the formulae satisfactory for union application which were extensively investigated:

1.	1.00% Pontacyl Fast Blue GB Extra Conc. 125%	CI 289
	0.15% Capracyl Orange R	--
	1.00% Pontamine Diazo Black OB Conc. 150%	Pr 147
	0.15% Pontamine Navy Blue BFN Conc. 150%	Pr 20
	Acetic Acid 1 hour at 212°F	
2.	0.16% Capracyl Yellow NW	--
	1.25% Capracyl Blue G	--
	1.00% Pontamine Diazo Black OB Conc. 150%	Pr 147
	0.15% Pontamine Navy Blue BFN Conc. 150%	Pr 20
	2.00% Triton X-100	
	Dye 1 hour at 212°F	
3.	1.25% Sulphon Cyanine BBA New	--
	0.10% Diamine Orange F	CI 459
	1.25% Oxydiaminogen OBA Conc. CF	Pr 147
	0.225% Benzo Chrome Black Blue Conc. CF	Pr 20
4.	1.75% Diazine Black DR	Pr 201
	2.50% Diazine Black S Extra	CI 317
	0.25% Diazine Blue BR 125%	Pr 74
	1.00% Fast Acid Blue CBI (Indocyanine B)	--
	1.40% Wool Fast Cyanone 3R 125%	CI 289
5.	3.00% Solantine Blue FF	Pr 71
	0.15% Solantine Orange 5RL	Pr 427
	0.15% Solantine Orange R	--
	1.50% Fast Wool Cyanone 3R	CI 289
	0.60% Fast Acid Brown RG	--
	0.40% Alizarin Violet R	CI 1080
	(After Treated)	

Formula 3 proved to be the best; it is the one recommended for Shade Blue 157. It was found to have the best lightfastness; this formula, however, is no better than the USAF Standard in wash and dry cleaning tests. Although better wash and dry cleaning fastness was sought, no formulation could be found which would improve these properties and still produce a union shade of adequate lightfastness. The deficiency lies primarily in the viscose dyes which can be used in union dyeing of nylon and viscose.

A supplier of the required fabric style could not be located; therefore, the plant dyeing phase of this fabric was cancelled.

III MANUFACTURE OF FABRICS

A. Shade Blue 84, Wool Serge 15 oz.

Considerable effort was expended to produce a length of fabric for USAF Shade Blue 84 which would closely match the USAF Standard under both daylight and artificial light conditions. Such results were obtained through the process of first manufacturing a series of test blanket fabrics with varying percentages of the fiber primaries, previously discussed, and also with small variations in the dye concentration on each of the primaries. The color of each test blanket was summarily evaluated spectrophotometrically, and the results obtained used in planning the next manufacturing trial.

Fourteen compositions were processed and evaluated prior to the production of the final LTIRF fabric based on test fabric found to be a good match of the standard under both daylight and artificial light. The LTIRF fabric satisfied the requirements, being well within color tolerances permitted in production deliveries.

With the exception of the problem of obtaining a close color match, no difficulty was experienced in the production dyeing and fabric manufacture. The importance of this shade and its critical color, however, necessitated the extensive experimentation mentioned. The narrow color tolerance allowable in the work under this contract would not be expected in fulfilling a regular production procurement; consequently, extended experimentation such as that undertaken herein should not be required to produce this fabric shade commercially. This is supported by the fact that thirteen out of the fourteen test pieces made in fabric development were judged to be commercially acceptable matches.

Tables 2, 3, 4, 5, 7, and 8 summarize the manufacturing data concerning fiber grade, dye formulae, blend composition, dyeing procedures, and the colors of fibers and fabrics.

B. Shade Blue 156, Wool Gabardine

The process of developing fabric for Shade Blue 156 was similar to that described for Shade Blue 84. Less work was necessary in this case, however, because a slightly greater color tolerance was acceptable and the shade change of Blue 156 in artificial light is not as great as it is in Blue 84.

A test blanket and a trial piece were manufactured to establish the correct shade. The daylight and artificial light colors of these products were evaluated visually and spectrophotometrically. It was considered that a satisfactory match had been attained; a 100-yard length of fabric was manufactured from the primaries specified in Table 3.

Tables 2, 3, 4, 5, 7, and 8 report the fiber grade, dye formulae, blend composition, dyeing instructions, and the colors of fibers and fabrics employed in the manufacture.

C. Shade Gray 167, Wool Gabardine

Manufacture of USAF Shade Gray 167 was fraught with difficulties. The real source of these difficulties lies in the extreme lightness of color of this fabric and, therefore, the very small percentage of dyes (0.04%) present. Under these conditions small differences in processing can result in large color variations. Trouble was encountered three times in the manufacture of this fabric. Twice numerous extraneous colored fibers (blue and black) quite obviously present in the fabrics had to be picked out by hand. When one piece was finished, a foreign yellow coloring matter was visible in the fabric, presumably the effect of an impurity in the water. This yellow was removed by scouring, but in scouring (the second scour on the one fabric) enough of the gray dye was removed to make the dye shade extremely light --- so light that the natural color of the wool was more prominent. The resultant fabric shade was therefore too light and too yellow.

Because of these difficulties, manufacture of the final yardage for this shade had to be undertaken a second time. In the second manufacture black fibers were again so prominent in the fabric that they had to be picked out by hand. Anticipated difficulties in the form of color loss by scouring after this "picking" operation were avoided by hand cleaning the fabric rather than scouring it by machine. This prevented a repetition of the loss in depth of shade encountered in the first trial, and a good match of the USAF Standard resulted.

The manufacturing data on fiber grade, dye formula, dyeing procedure, and the colors of fibers and fabric are summarized in Tables 2, 3, 5, 7, and 8.

D. Shade Tan 193, Wool Gabardine Type IV

Since this fabric is a solid shade, relatively easy control of color prediction based on spectrophotometric measurement of the test blanket and its component-dyed sliver was thought possible. Only one blanket was made. Its color was measured and found to be reasonably close to the standard but not a satisfactory match. The results of color measurement, that is, the change in shade in going from sliver to fabric, were used to estimate or predict the sliver color necessary to yield a satisfactory fabric match to the USAF Standard. Sliver was dyed to the estimated color and the LTIRF fabric was made from this sliver. The resultant fabric, although acceptable, did not match the USAF Standard as well as expected. This appears due to processing variations which can produce appreciable color modification when the shade is as light as that of this fabric (0.09% dye). Spectrophotometric measurements show the greatest difference in color to be in the brightness value. The LTIRF fabric is different from the standard primarily because of its lightness of shade. Since the dyed sliver before fabric manufacture was adequately heavy, it appears more dye was lost during processing of the LTIRF fabric than was lost by the test blanket.

Tables 2, 3, 5, 7, and 8 list the fiber grade, dye formula, dyeing procedure, and the colors of fibers and fabrics employed in the manufacturing phase.

IV TEST RESULTS

A. Fiber Composition

As specified in Table 2, 64's grade Australian wool was used in all four of the 100-yard length fabrics manufactured. The percentage composition of each of the fiber primaries used in Shade Blue 84 and Shade Blue 156 (blended fiber fabrics) are specified in Table 4. The dyeing formula for each primary, both the laboratory formula and the actual production formula, is given in Table 3, and the color specifications for pads of each fiber primary are given in Table 7.

B. Color

The colors of the 100-yard length of each LTIRF fabric and the USAF Standards were measured on a General Electric Recording Spectrophotometer using MgO as a standard white. Spectrophotometric measurement procedures are described in Table 6 and their principles are discussed in the Appendix.

The trichromatic coefficient measurements for each of the fabrics are given in Tables 7 and 8. These results are plotted graphically in relation to color tolerance ellipses in Figures 1-6.

Figures 1 and 2 also show the colors of the fibers used in blending to produce the "mixy" appearance of Shade Blue 84 and Shade Blue 156.

Shade Blue 84, Figure 3, is within a 2.5 MacAdam color tolerance ellipse previously selected as a desired goal for the project.

Shade Blue 156, Figure 4, is a little outside 2.5 color tolerance ellipse, but it was an acceptable match to the Air Force.

Shade Gray 167, Figure 5, is within a 1.5 color ellipse and is acceptable although it is slightly lighter (higher brightness value) than the standard.

Shade Tan 193, Figure 6, although within a 1.5 color tolerance ellipse, is noticeably lighter (high brightness (Y) value) and was judged just barely acceptable.

C. Colorfastness Properties

All LTIRF fabrics were tested for colorfastness to light, crocking, perspiration dry cleaning, and laundering. The test procedures employed are described in Table 9.

All fabrics showed good fastness to crocking, perspiration, dry cleaning, and laundering. They completely satisfy the requirements for USAF specifications on thes fabrics. These results are shown in Table 12.

Considerable emphasis was placed on a detailed study of the colorfastness to light properties because of the reported greater need for improvement in this property than in any other. Samples of the USAF Standard and the LTIRF fabric were tested simultaneously in the Fadeometer for successive 20-hour intervals to determine the difference in fastness of the two fabrics exposed to the same environment. Account was taken of the time required to produce a visual break, and, later, the color changes produced at selected 20-hour intervals were determined spectrophotometrically.

The color differences produced by the fading of each fabric are presented in Table 10. These results are also illustrated in Figures 7-10.

The extensive investigation of Shade Blue 84, Table 11 and Figure 7, clearly showed that the LTIRF fabric is markedly superior to the USAF Standard. The LTIRF fabric shows color differences of 0.86 and 1.08 at 80 and 140 hours as compared with 1.70 and 2.54 respectively for the USAF Standard. Thus, there is less color change in the LTIRF fabric after 140 hours than in the standard after 80 hours. Visually the standard was judged to break at 60 hours while the LTIRF fabric broke only after 140 hours.

The effect on the lightfastness of varying dye lots and fiber blend compositions was studied by testing the fastness of seven different test blankets manufactured as part of the color control program. As was expected, varying the composition does affect lightfastness, but the effect on the LTIRF formula was small. The greatest fade was 1.91 color difference units after 160 hours exposure, and this fade, the worst shown by the blankets, is about equal to that produced by 80 hours exposure of the USAF Standard. Thus, very reliable and very resistant fastness is provided by the LTIRF formula.

Great improvement in the fastness of Shade Blue 156 is shown in Table 10 and Figure 8. A perceptible break (1.02) is produced in the LTIRF fabric after 140 hours of Fadeometer exposure. The USAF Standard shows a greater color difference (2.23) than this at 80 hours. It is estimated that 40 hours on the standard produces as much color change as 140 hours on the LTIRF fabric. Since the dye components in Shade Blue 156 are the same as those in Shade Blue 84, its fading properties under varying production conditions should be as reliable as those of Shade Blue 84.

The data on the fading of Shade Gray 167 presented in Table 10 and plotted in Figure 9 show that a tremendous improvement has been made in the lightfastness of this shade. The color changes produced at 140 hours are of the greatest significance; the LTIRF fabric appears a little lighter, while the color of the standard appears almost completely destroyed and is a yellow-green rather than a gray. Table 10 shows 140-hour color differences of 1.92 for the LTIRF fabric and 6.17 for the standard. Exposure time for a visual break has been increased from 40 hours with the standard to 60 hours with the LTIRF fabric. Significantly lower color differences are also shown by the 20-, 40-, 60-, and 80-hour samples.

The value of a low rate of fade on long exposures is strikingly obvious in the data on Shade Gray 167. It should be noted that the fade does not increase on going from the 80- to the 140-hour exposures. This is very similar to the case of the long exposures for the LTIRF Shade Tan 193 and is believed to be a very important property for any fabric as light in color as Shade Gray 167 and Shade Tan 193. Since the concentration of dye present is so small (Shade Gray 167 = 0.04%, Shade Tan 193 = 0.09%), it is extremely unlikely that a perceptible fade in 40 to 60 hours can be avoided. Actually, the yellowing of wool by light is sufficient to cause a perceptible fade. However, if the rate of fade after the initial change can be kept low --- provided, of course, this initial change is moderate --- then the optimum fastness from the point of view of wearability will be attained. This is believed to be the condition achieved for both Shade Gray 167 and Shade Tan 193 even though it is believed the dyestuff formula used in Shade Tan 193 is capable of greater fastness than that obtained in the project.

The lightfastness of the LTIRF fabric for Shade Tan 193 was found to be a little inferior to that of the USAF standard. Data showing this are given in Table 10 and Figure 10. Although the LTIRF fabric fades more rapidly during the 0- to 80-hour periods, the fading after the longer exposure of 140 hours is at least equal to the USAF Standard and may even be slightly superior. It can be seen in Table 10 that the rate of fading of the LTIRF fabric is very small after 80 hours --- a 0.11 color difference increase between 80 and 140 hours --- while the USAF Standard rate of fade continues large (a 0.80 color difference increase). Therefore, it is likely that after two hundred or more hours the LTIRF fabric would be superior.

The initial laboratory work with Shade Tan 193 indicated that the LTIRF formula would at least equal and possibly surpass slightly the lightfastness of the USAF Standard. It is interesting to speculate concerning why the fabric finally produced with this formula did not equal the laboratory estimates. Although it cannot be definitely substantiated, it can be postulated with sound reasoning that the source of the lower than anticipated fastness is a function of the depth of color attained on the LTIRF fabric. It has already been pointed out that the major difference between the color of the standard and that of the LTIRF fabric was the brightness value.

Here, brightness, an inverse measure of color intensity or depth, is appreciably greater in the case of the LTIRF fabric. This means that the LTIRF fabric is lighter than the standard. It is a well known fact that lightfastness decreases markedly with increasing lightness of shade. This factor is considered to be the cause of the lower than expected fastness of the LTIRF fabric. The colorfastness properties of this fabric fulfil a situation similar to that presented in the discussion of variations in compositions of the Shade Blue 84 blanket fabrics, but, because Shade Tan 193 is much lighter than Shade Blue 84, the effect of this phenomenon on Shade Tan 193 is more serious.

The laboratory dyeing evaluations of the fastness properties of Shade Sage Green 511 and Shade Blue 157 have already been discussed. Since no fabric was manufactured the laboratory conclusions are the only ones that can be drawn. Good wetfastness properties and improved lightfastness for Shade Sage Green 511 were obtained. No marked improvement in lightfastness and wetfastness of Shade Blue 157 could be discovered.

D. Physical Properties

The four LTIRF manufactured fabrics were subjected to a series of tests of their physical properties as called for by the appropriate Government Specifications listed in Table 1. Ends and Picks, Weight, Breaking Strength, Air Permeability, Sponging Shrinkage, and pH were tested in accordance with Specification CCC-T-191 test methods. These tests are described in Table 13. The tests were made to determine the conformance of LTIRF fabrics to the specifications and were not directive in nature.

The test results obtained with all fabrics are presented in Table 14. Small deviations from the government specifications of these fabrics were noted in a few cases. These consisted of three picks per inch difference for Shade Blue 84; 0.62%, 4.20%, 3.73% sponging shrinkage differences for Shade Blue 84, Shade Gray 167, and Shade Tan 193 respectively; and 0.1, 0.8, 0.9, and 0.9 pH differences for Shades Blue 84, Blue 156, Gray 167, and Tan 193 respectively. It is presumed that these differences arose from the manufacturers' attempts to maintain color control of the fabrics by controlling the time in various operations such as finishing so as to reproduce the color conditions of the trial blankets. Thus, the control for physical dimensions were not as stringently exercised as would be the case in the production of a normal procurement fabric. These deviations, however, are not considered serious limitations on the quality of the fabrics of this project.

TABLE 1

OBJECTIVES OF PROJECT AND USAF FABRIC REQUIREMENTS

Cloth and Shade	Specification	Research: Dyeing	Research: Manufacture	Colorfastness Requirements
Wool, Serge 15 oz. Shade Blue 84	MIL-C-849 dtd 28 July '49	Develop dye formulation of greater color-fastness	100 yards of fabric to match standard but with greatest colorfastness	CCC-T-191 Test Method Light = 80 hours Good fastness to laundering, water, crocking, perspiration, and dry cleaning.
Wool, Gabardine Shade Blue 156	MIL-C-10176A dtd 26 Jan. '51	"	"	"
Wool, Gabardine Shade Gray 167	MIL-C-6403 dtd 26 Jan. '51	"	"	"
Wool, Gabardine Shade Tan 193	JAN-C-391 dtd 30 Aug. '46 amendment dated 30 Jan. '51	"	"	"
Nylon, Satin Shade Sage Green 511	16211 dtd 2 June '49	"	None	CCC-T-191 Test Method Light = 40 hours Others to meet specifications
Nylon, Rayon Shade Blue 157	MIL-C-4072 dtd 21 March '50	"	None	"

TABLE 2

FIBER SPECIFICATIONS

Fibers in LTIRF Fabrics	
Wool Serge Shade Blue 84	64s Grade Australian Wool
Wool Gabardine Shade Blue 156	64s Grade Australian Wool
Wool Gabardine Shade Gray 167	64s Grade Australian Wool
Wool Gabardine Shade Tan 193	64s Grade Australian Wool

TABLE 3

DYE FORMULATIONS FOR LTIRF FABRICS

Fabric	Laboratory Formula	Production Formula	Dyestuffs
Wool Serge Blue 84			
Primary No. 1 - Blue	2.5%	--	Helindon Blue B (Indigo) CI 1177
	--	5.0%	Algosol Blue O CI 1178
Primary No. 2 - Blue	2.1%	2.2%	Acid Chrome Blue RRA Pr 7
Primary No. 3 - Gray	0.315%	0.320%	Alizarine Light Grey BBLW Pr 206
	0.080%	0.067%	Anthraquinone Violet 3RA CI 1080
Wool Gabardine Blue 156			
Primary No. 1 - Blue	2.0%		Helindon Blue B (Indigo) CI 1177
		4.2%	Algosol Blue O CI 1178
Primary No. 2 - Blue	2.0%	1.9%	Acid Chrome Blue RRA Pr 7
Primary No. 3 - Gray	0.315%	0.320%	Alizarine Light Grey BBLW Pr 206
	0.080%	0.067%	Anthraquinone Violet 3RA CI 1080
Wool Gabardine Gray 167			
	0.022%	0.0247%	Alizarine Light Gray BBLW Pr 206
	0.016%	0.0130%	Anthraquinone Violet 3RA CI 1080
Wool Gabardine Tan 193			
	0.046%	0.0258%	Chrome Fast Orange 3RLA Pr 247
	0.042%	0.0202%	Alizarine Light Gray BBLW Pr 206
	0.003%	--	Anthraquinone Violet RA CI 1080
		*0.0035%	Fast Flavine RA Pr 1

* Not specified in LTIRF formula.

TABLE 4

DYED FIBER COMPONENTS IN LTIRF FABRICS

LTIRF FABRICS	PERCENTAGE COMPOSITION
Wool Serge Blue 84	
Primary No. 1 (Helindon Blue B)	33%
Primary No. 2 (Blue RRA)	35%
Primary No. 3 (Gray BBLW)	32%
Wool Gabardine Blue 156	
Primary No. 1 (Helindon Blue B)	50%
Primary No. 2 (Blue RRA)	38%
Primary No. 3 (Gray BBLW)	12%
Wool Gabardine Gray 167 (Solid Shade)	100%
Wool Gabardine Tan 193 (Solid Shade)	100%

TABLE 5

PROCEDURES FOR DYEING

FORMULAE FOR SHADE BLUE 84

Primary No. 1 - Indigo Blue

10.0% Indigo 20% Paste (CI 1177)
or
2.5% Helindon Blue B Conc. Pdr.

Indigo Vat	.09 oz. per gallon (based on total volume dye liquor) Caustic Soda Dry Heat to 120°F Sprinkle in .12 oz. per gallon (based on total volume dye liquor) Hydro Conc. Vat above in 7% to 10% of total liquor to be used.
Spring Bath	Add wool and wet out .20 oz. per gallon (based on total liquor) Ammonia 3.00 oz. per gallon (based on total liquor) Common Salt Sprinkle in dry .12 oz. per gallon (based on total liquor) Hydro Conc.
Add Dye	Add all vatted indigo prepared as above, at 90°-100°F Heat to 110-120°F and hold 30 minutes Add without washing 1% Ammonium Sulphate (on weight of wool) Add without washing 2 times as much Bicarbonate as Caustic Soda used, or .18 oz. per gallon Run 10 minutes and wash well Add .12% (on weight of wool) Peroxide 120 Vol. 15 minutes at 100°F Wash Acidify - 3.00% Acetic Acid 28% (on weight of wool) at 150°F Wash

Primary No. 2 - Blue RRA

2.1% Acid Chrome Blue 2RA (Pr. 7)
Dyed Topchrome using:
10% Glaubers Salt
1-1/2% Acetic Acid 56%
Boil 30 minutes
1 - 2% Sulphuric Acid
Boil 30 minutes
Add 1-1/4% Bichromate of Soda
Boil 45 minutes to 60 minutes

Table 5 (continued)

Primary No. 3 - Gray

0.315% Alizarine Light Gray BBLW
0.08% Anthraquinone Violet 3RA
.25% Bichromate of Soda
10.00% Glaubers Salt
1.50% Acetic Acid 28%
Boil 30 minutes and add
1.50% Acetic Acid 28% (if necessary)
Boil 30 minutes longer

FORMULAE FOR SHADE BLUE 156

Procedures same as those reported above under Blue 84.
Percentage of dyestuffs as reported in Table 2.

FORMULA FOR SHADE GRAY 167

0.022% Alizarine Light Gray BBLW
0.016% Anthraquinone Violet 3RA
10% Ammonium Acetate
3% Sodium Bichromate
Boil 3/4 hour
Exhaust with 1-2% Acetic Acid 56% (if necessary)
Boil 1/2 hour

FORMULA FOR SHADE TAN 193

0.046% Chrome Fast Orange 3RLA Pr 247
0.042% Alizarine Light Gray BBLW Pr 206
0.003% Anthraquinone Violet RA CI 1080
10% Ammonium Acetate
3% Chromate Mordant
Boil 3/4 hour
Exhaust with 1-2% Acetic Acid 56% (if necessary)
Boil 1/2 hour
Wash

TABLE 6

COLOR MEASUREMENT TEST PROCEDURES

Spectrophotometric measurements of the color of textile materials were employed in this project for two purposes. First, during the development of the specified fabrics, these measurements were used as a color control for dyeing and fabric manufacture. Second, as part of the testing program, the color of LTIRF fabrics and the USAF Standard fabrics were numerically and graphically specified by means of their trichromatic coefficients.

All measurements were made on a General Electric Recording Spectrophotometer containing an automatic tristimulus integrator. Four measurements were made on different areas of each specimen. The results reported are the mean values of these four measurements.

During the testing of the final LTIRF fabrics and the USAF Standard fabrics, these measurements were made using Magnesium Oxide as the standard white. All specimens of one shade were measured at the same time to minimize instrumental variations.

When measurements were made for the purpose of color control, Magnesium Carbonate or Vitrolite was used as a standard white.

Trichromatic coefficients were calculated in the manner reported by Hardy (1). Color differences were calculated in the manner described by Adams (2) and Nickerson (3), and the graphical plotting of trichromatic coefficients and color tolerance ellipses were carried out according to the work of MacAdam (4), Davidson and Godlove (5), and Davidson and Haire (6).

A discussion of spectrophotometry and colorimetry applicable to the color control work of this project is presented in the Appendix.

TABLE 7

SPECTROPHOTOMETRIC MEASUREMENTS OF LTIRF FABRICS
AND FIBER COMPONENTS

Fabrics	Trichromatic Coefficients		
	x	y	Y
Wool Serge, Shade Blue 84			
Fabric	.2630	.2525	.0464
Fiber Primary No. 1	.2334	.2158	.0214
Fiber Primary No. 2	.2635	.2280	.0244
Fiber Primary No. 3	.2866	.2913	.1548
Blend of Fiber Primaries	.2667	.2578	.0482
Wool Gabardine, Shade Blue 156			
Fabric	.2536	.2386	.0302
Fiber Primary No. 1	.2258	.2091	.0222
Fiber Primary No. 2	.2646	.2316	.0200
Fiber Primary No. 3	.2881	.2968	.1547
Blend of Fiber Primaries	.2404	.2366	.0317
Wool Gabardine, Shade Gray 167			
Fabric	.3189	.3231	.3930
Fiber	.3185	.3231	.4212
Wool Gabardine, Shade Tan 193			
Fabric	.3425	.3465	.2968
Fiber	.3451	.3500	.3284

TABLE 8

SPECTROPHOTOMETRIC MEASUREMENTS OF USAF STANDARDS AND LTIRF FABRICS

Fabrics	Trichromatic Coefficients		
	x	y	Y
Wool Serge, Shade Blue 84			
USAF Standard	0.2625	0.2535	0.0470
LTIRF Fabric	0.2630	0.2525	0.0464
Wool Gabardine, Shade Blue 156			
USAF Standard	0.2510	0.2343	0.0290
LTIRF Fabric	0.2536	0.2386	0.0302
Wool Gabardine, Shade Gray 167			
USAF Standard	0.3184	0.3236	0.3880
LTIRF Fabric	0.3189	0.3231	0.3930
Wool Gabardine, Shade Tan 193			
USAF Standard	0.3436	0.3482	0.2803
LTIRF Fabric	0.3425	0.3465	0.2968

TABLE 9

COLORFASTNESS TEST PROCEDURES

Colorfastness to Light

Colorfastness to light tests were performed in accordance with Specification CCC-T-191.

Test specimens, mounted in an open back type of holder, were exposed to the light of an Atlas FDR-A Fadeometer for 80- and 140-hour periods. A 110°F temperature was maintained. The Bureau of Standards "Standard Fading Paper" was used to calibrate the exposure hours.

Test specimens were examined after each 20 hours of exposure and the exposure time to produce a visual break in color (as judged by testing technician) was noted.

The color of unfaded and faded samples was measured on a G. E. Recording Spectrophotometer. The color changes produced by fading were shown graphically by plotting the trichromatic coefficients of faded and unfaded samples and were recorded numerically by calculating the color difference of fading according to the Adams formula.

Since the rate of fading of Shade Blue 84 and Shade Blue 156 was low, only the 80- and 140-hour specimens were measured on the G. E. Recording Spectrophotometer. The 20-, 40-, 60-, 80-, and 140-hour specimens of the more rapidly fading Shade Tan 193 and Shade Gray 167 shades were measured.

Colorfastness to Crocking

Wet and dry tests of crocking were performed in accordance with Specification CCC-T-191b, Method 5650. The results obtained were rated as follows:

Good - No appreciable staining of the wet white multifiber test cloth.

Fair - Appreciable staining of wet white multifiber test cloth but no appreciable staining of dry white multifiber test cloth.

Colorfastness to Perspiration

Acid and Alkaline Perspiration Tests were made in accordance with Specification CCC-T-191b, Test Method 5682. A rating of "good" indicates no change in shade of the test specimen, no migration of color, and no staining of white multifiber test cloth.

Colorfastness to Dry Cleaning

(a) Dry cleaning tests were performed in accordance with Specification CCC-T-191a, Method 5620. A rating of "good" indicates no change in shade, no migration of color, and no staining of multifiber test cloth.

TABLE 9 (continued)

(b) Dry and Wet Dry Cleaning Tests were made in accordance with Commercial Standard CS59-44. Class 4 shows no appreciable staining of the composite test fabric.

Colorfastness to Laundering

Tests were made in accordance with CCC-T-191b, Method 5614 ($100^{o}F$).

TABLE 10

FADING TEST RESULTS

Fabrics	Color Differences (Adams Units) 40 Hour Fade	60 Hour Fade	80 Hour Fade	140 Hour Fade	Exposure Time to Color Break (Hours)
Wool Serge, Shade Blue 84					
USAF Standard	--	--	1.70	2.54	60
LTIRF Fabric	--	--	0.86	1.08	140
Wool Gabardine, Shade Blue 156					
USAF Standard	--	--	2.23	2.83	40
LTIRF Fabric	--	--	0.70	1.02	140
Wool Gabardine, Shade Gray 167					
USAF Standard	1.698	2.284	2.742	6.166	40
LTIRF Fabric	1.064	1.764	2.008	1.920	60
Wool Gabardine, Shade Tan 193					
USAF Standard	1.36	1.47	1.86	2.66	40
LTIRF Fabric	1.81	2.22	2.57	2.68	40

TABLE 11

FADING TEST RESULTS OF WOOL SERGE, SHADE BLUE 84

Fabrics	Exposure Time to Color Break (Hours)	COLOR DIFFERENCE (Adams Units) 80-Hour Exposure LTIRF	80-Hour Exposure *Textile Aniline	160-Hour Exposure LTIRF	160-Hour Exposure *Textile Aniline
USAF Standard	60-80	1.700	1.620	2.540	2.370
LTIRF Fabric	140	0.860	0.704	1.080	--
Blanket No. 1	100	0.560	--	1.910	--
Blanket AA	140	1.230	1.000	1.250	1.320
Blanket BB	100-120	1.160	0.840	1.840	1.380
Blanket CC	120	0.920	0.760	1.470	1.150
Blanket LL	140	1.660	1.630	1.880	1.690
Blanket RR	140	0.720	1.108	1.040	1.430

* Conducted by this organization on their Fadeometer to corroborate the data obtained with the LTIRF Fadeometer.

TABLE 12

COLORFASTNESS TEST RESULTS

	Wool Serge Shade Blue 84	Wool Gabardine Shade Blue 156	Wool Gabardine Shade Gray 167	Wool Gabardine Shade Tan 193
Laundering				
CCC-T-191a (100°F Test)	No stain of composite test fabric. No color loss. No shade alteration.	No stain of composite test fabric. No color loss. No shade alteration.	No stain of composite test fabric. No color loss. No shade alteration.	No stain of composite test fabric. No color loss. No shade alteration.
Dry Cleaning				
CCC-T-191b (Test Method 5620)	Good	Good	Good	Good
CS59-44				
Wet Test	Class 4	Class 4	Class 4	Class 4
Dry Test	Class 4	Class 4	Class 4	Class 4
Crocking				
Wet Test	Fair	Good	Good	Good
Dry Test	Good	Good	Good	Good
Perspiration				
Acid Test	Good	Good	Good	Good
Alkaline Test	Good	Good	Good	Good

TABLE 13

PHYSICAL TEST PROCEDURES

Ends and Picks per Inch

These were determined in accordance with Specification CCC-T-191b, Method 5050. The results are the means of two count determinations.

Weight Determination

Seventy-two square inches of material were weighed on an analytical balance. Fabric weight in oz/yd^2 was calculated from this determination.

Breaking Strength

These tests were performed in accordance with Specification CCC-T-191b, Test Method 5100, Grab Method. The results reported are the mean values for five warp and five filling tests.

Tear Strength

Tear strength measurements were made in accordance with Specification CCC-T-191b, Test Method 5134, Tongue Method. The results reported are the mean values for five warp and five filling tests.

pH of Water Extract

Acidity (pH) of fabrics, potentiometric method; tests were made in accordance with Specification CCC-T-191b, Method 2811, modified as follows. Measurements were made directly in 250 ml glass stoppered Erlenmeyer flasks instead of the 100 ml beakers specified in the test procedure. This modification became necessary when it was found that absorption of Carbon Dioxide from the air lowered the pH of the water solutions in a beaker from 0.6 to 1.0 pH units. The distilled water used for these tests had a pH of 6.4.

Air Permeability

Air Permeability Tests were made in accordance with Specification CCC-T-191b, Test Method 5450.

Shrinkage in Sponging

Fabric shrinkage in sponging tests were performed in accordance with Specification CCC-T-191b, Method 5590.

TABLE 14

PHYSICAL TEST RESULTS

Properties	Wool Serge Shade Blue 84	Wool Gabardine Shade Blue 156	Wool Gabardine Shade Gray 167	Wool Gabardine Shade Tan 193
Ends per Inch	70.0	111.0	84.5	84.5
Picks per Inch	55.0	57.0	51.0	51.2
Weight (oz/yd^2)	9.35	9.92	7.84	7.08
Air Permeability ($ft^3/ft^2/min$)	15.5	18.1	26.4	39.5
Breaking Strength (lbs)				
Warp	122.6	162.2	138.6	112.8
Filling	95.6	65.7	74.4	55.8
pH of Water Extract	3.9	8.8	8.9	8.9
Tear Strength (lbs)				
Warp	6.0	5.8	4.7	4.4
Filling	5.3	3.1	3.6	3.4
Shrinkage in Sponging (%)				
Warp	5.62	4.53	8.20	8.73
Filling	0.00	1.00	0.40 S*	1.13

* S designates stretch instead of shrinkage.

TABLE 15

DYESTUFFS EVALUATED FOR SHADE BLUE 84 AND SHADE BLUE 156

BLUE	
Acid Chrome Blue RRA	Pr 7
Mordant Blue B	Pr 93
Milling Blue BL	--
Anthracene Navy Blue SWGG	CI 1059
Anthracene Navy Blue SWR	CI 1062
Chromidigen Blue BRA Conc	Pr 411
Indigo	CI 1177
Helindon Blue B	CI 1177
Algosol Blue O-CF	CI 1178
Algosol Blue 06B-CF	CI 1185
Indigo Vat #1	CI 1177
Helindon Red BBW Paste	--
Helindon Brown RRD Pdr.	Pr 121
Helindon Brilliant Blue 4B Conc. Pdr.	CI 1184
Monochrome Black Blue GA CF	Pr 299
Metomega Chrome Cyanine BLL or Alizarole Cyanone BLL	--
GRAY	
Algosol Gray IBL-CF	--
Indigo	CI 1177
Indanthrene Brown RRD	Pr 121
Helindon Pink R	Pr 109
Alizarine Light Gray BBLW	Pr 206

TABLE 15 (continued)

Anthraquinone Violet 3RA	CI 1080
Helindon Blue B Conc. Pdr.	CI 1177
Helindon Brilliant Blue 4B Conc. Pdr.	CI 1184
Helindon Red BBW Paste	--
Helindon Brown RRA Paste	Pr 121
Anthraquinone Violet D	CI 1080

TABLE 16

DYESTUFFS EVALUATED FOR SHADE GRAY 167

Alizarine Fast Gray BBLW	Pr 206
Anthraquinone Violet 3RA	CI 1080
Capracyl Gray GN	--
Capracyl Yellow NW	--
Capracyl Red BB	--
Capracyl Yellow NW	--
Capracyl Violet R	--
Anthraquinone Green GN	CI 1078
Acid Alizarine Brown RLL	--
Algosol Gray IBL	--
Algosol Brown IBR	Pr 188
Algosol Pink IR	Pr 109

TABLE 17

DYESTUFFS EVALUATED FOR SHADE TAN 193

Chrome Fast Orange 3RLA	Pr 247
Alizarine Light Gray BBLW	Pr 206
Anthraquinone Violet RA	CI 1080
Pontachrome Yellow FR	--
Pontachrome Orange L	--
Capracyl Gray GN	--
Acid Alizarine Flavine RA Extra	Pr 1
Monochrome Red FGA Conc.	Pr 300
Chrome Fast Orange 3RLA	Pr 247
Algosol Brown IBR	Pr 188
Algosol Gray IBL	--
Algosol Golden Orange IRR	--
Algosol Olive Green IB	Pr 293

TABLE 18

DYESTUFFS EVALUATED FOR SHADE SAGE GREEN 511

Capracyl Yellow 3RD	--
Capracyl Orange R	--
Anthraquinone Green CN	CI 1078
Acetamine Fast Yellow 4RL	--
Celanthrene Fast Pink 3B	--
Acetamine Scarlet B	Pr 244
Celanthrene Brilliant Blue FFS Conc. 200%	Pr 228
Ponsol Olive GGL Paste	--
Ponsol Direct Black 3G Dbl. Paste	--
Celliton Fast Blue FFRN Ex. Conc.	--
Celliton Fast Yellow 4RL Conc.	--
Celliton Fast Pink RFD	--
Palatine Fast Blue BNDA	Pr 318
Palatine Fast Orange GENA	Pr 315
Palatine Fast Yellow GRNA	Pr 316
Alizarine Fast Gray BLN	Pr 206
Acid Alizarine Brown RLL	--
Supranol Yellow RA	CI 642
Carbanthrene Gray GFL Paste	--
Carbanthrene Olive T Paste	--
Carbanthrene Brilliant Green Dbl Paste	CI 1101
Chromolan Blue NGG	Pr 144
Alizarine Cyanone Green GN	CI 1078
Chromolan Orange GN	Pr 315
Chromolan Red 3RB	--

TABLE 19

DYESTUFFS EVALUATED FOR SHADE BLUE 157

Pontacyl Fast Blue GB Extra Conc. 125%	CI 289
Capracyl Orange R	--
Pontamine Diazo Black OB Conc. 150%	Pr 147
Pontamine Navy Blue BFN Conc. 150%	Pr 20
Capracyl Yellow NW	--
Capracyl Blue G	--
Sulphon Cyanine BBA New	--
Diamine Orange F	CI 459
Oxydiaminogen OBA Conc.	Pr 147
Benzo Chrome Black Blue Conc.	Pr 20
Diazine Black DR	Pr 201
Diazine Black S Extra	CI 317
Diazine Blue BR 125%	Pr 74
Fast Acid Blue CBI	--
Wool Fast Cyanone 3R 125%	CI 289
Solantine Blue FF	Pr 71
Solantine Orange 5RL	Pr 427
Solantine Orange R	--
Fast Wool Cyanone 3R	CI 289
Fast Acid Brown RG	--
Alizarine Violet R	CI 1080

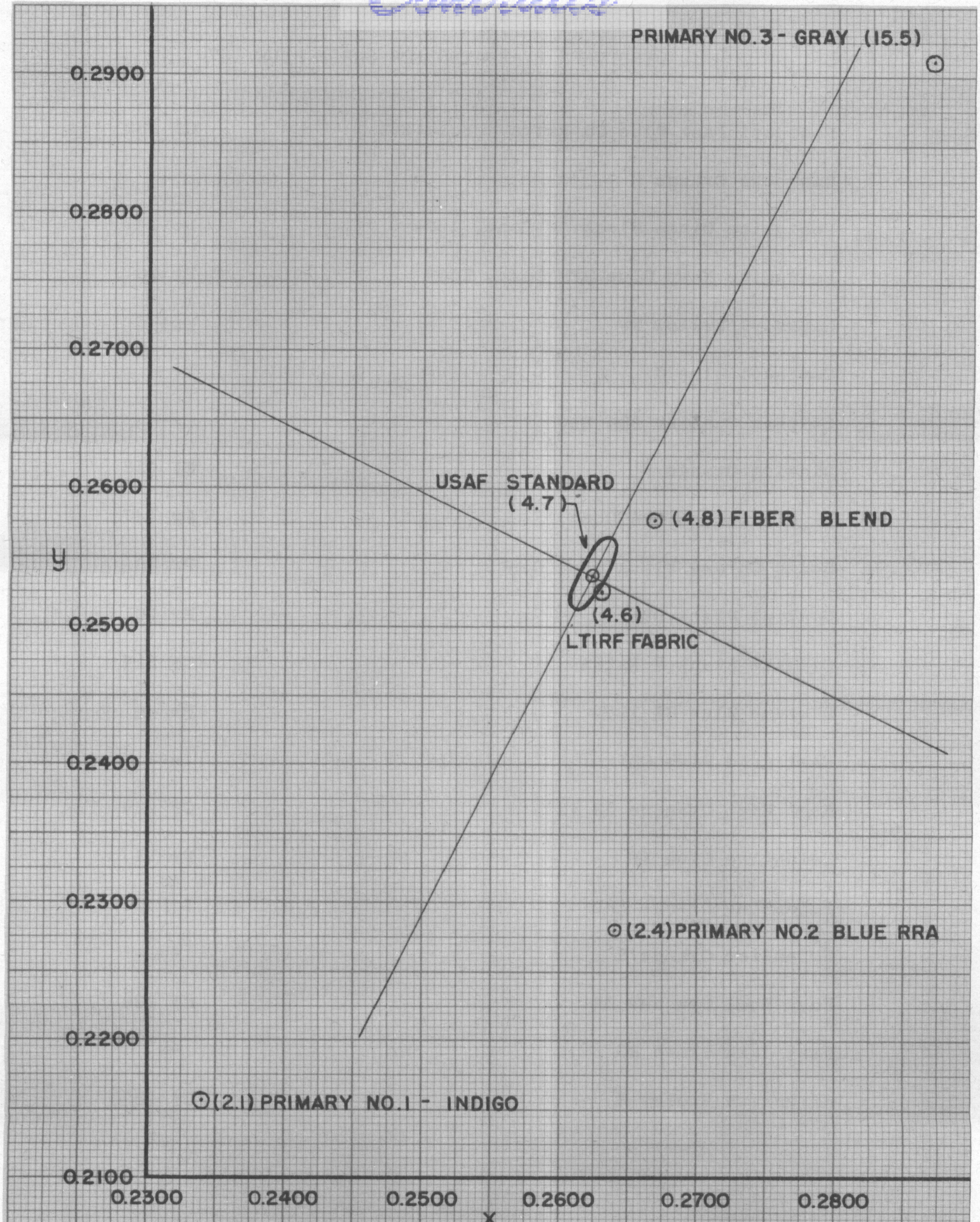

FIGURE 1. CHROMATICITY DIAGRAM - USAF WOOL SERGE SHADE BLUE 84 - FIBER COMPONENTS

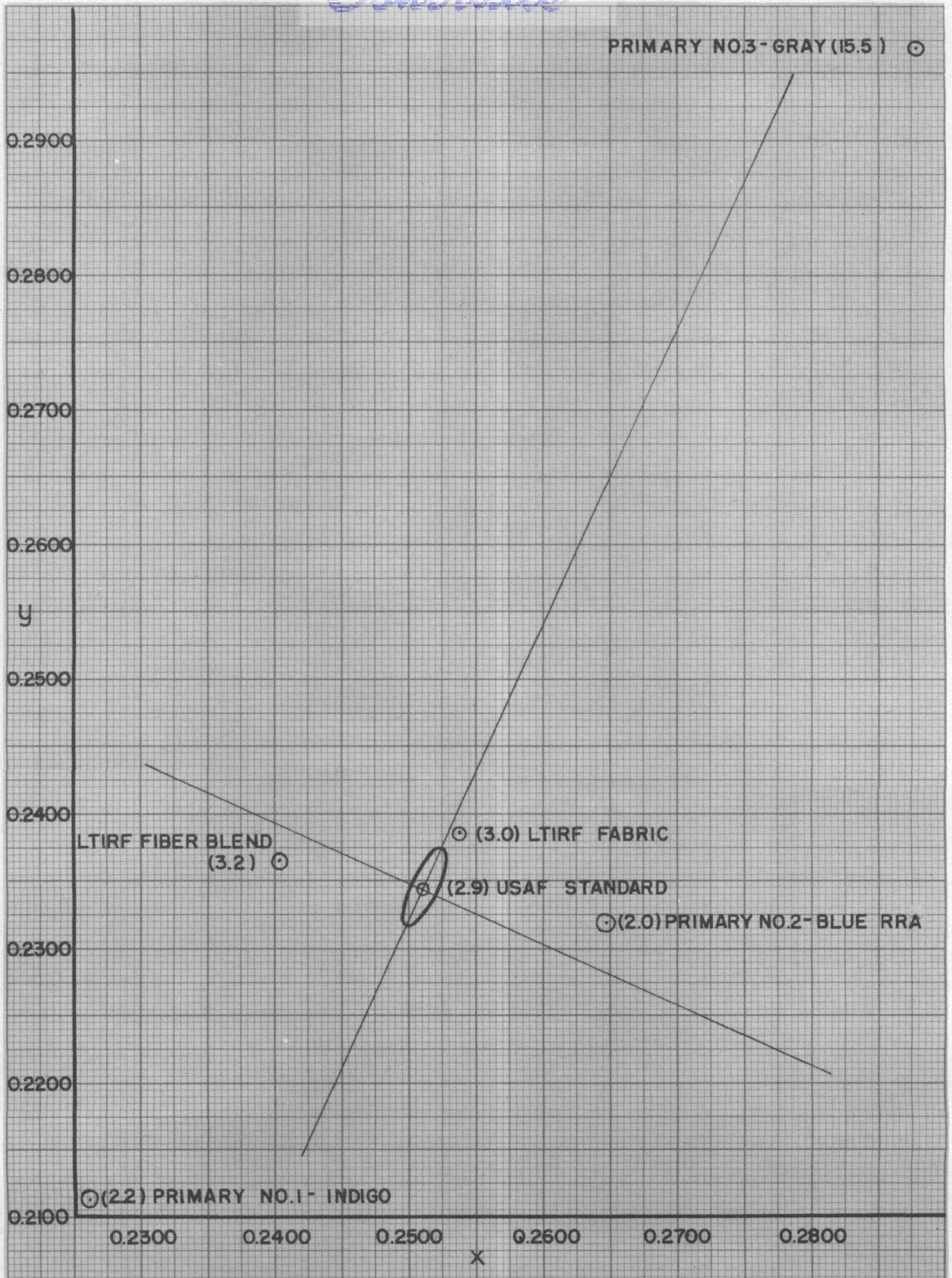

FIGURE 2. CHROMATICITY DIAGRAM - USAF WOOL GABARDINE SHADE BLUE 156 - FIBERS

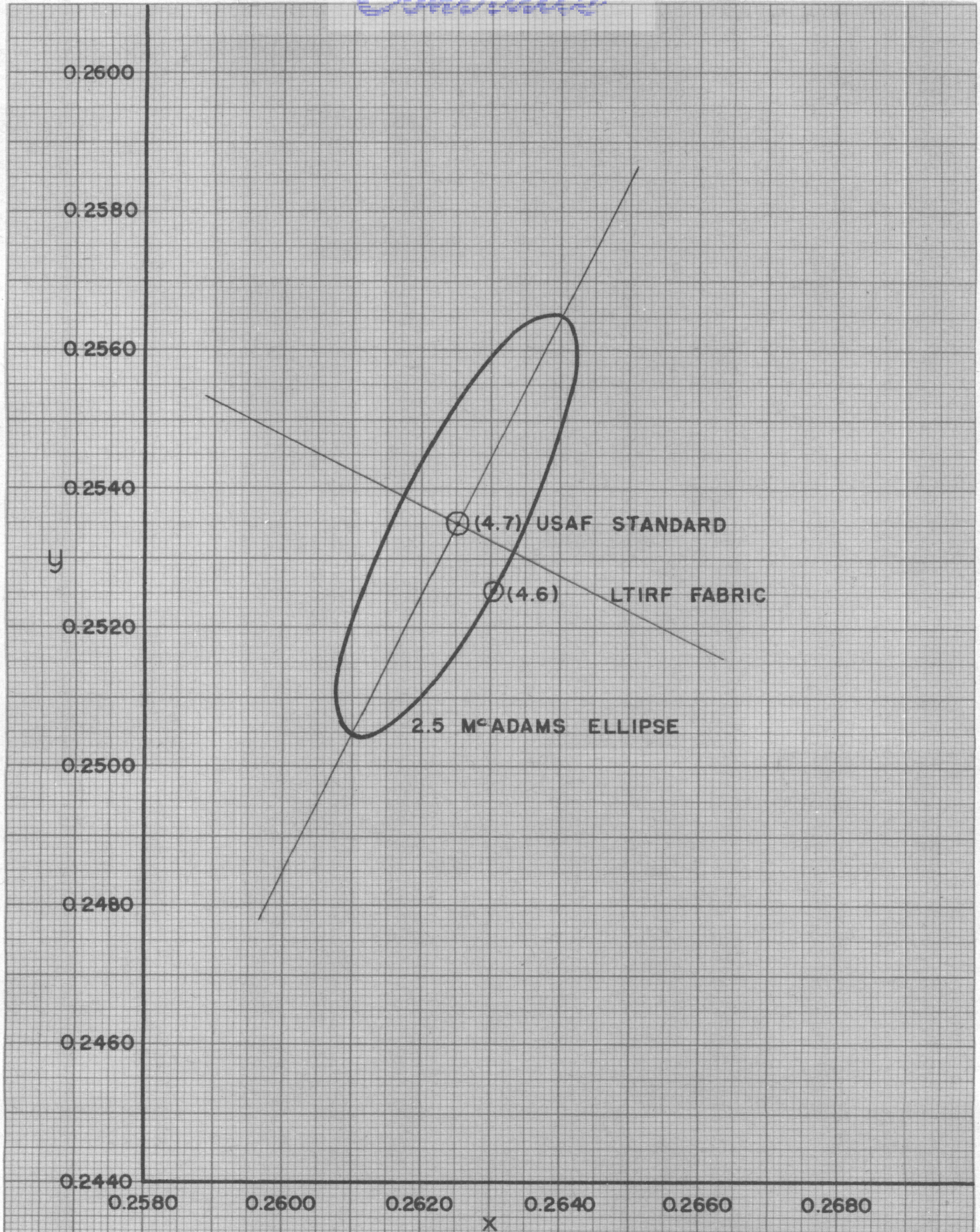

FIGURE 3. CHROMATICITY DIAGRAM - USAF WOOL SERGE SHADE BLUE 84

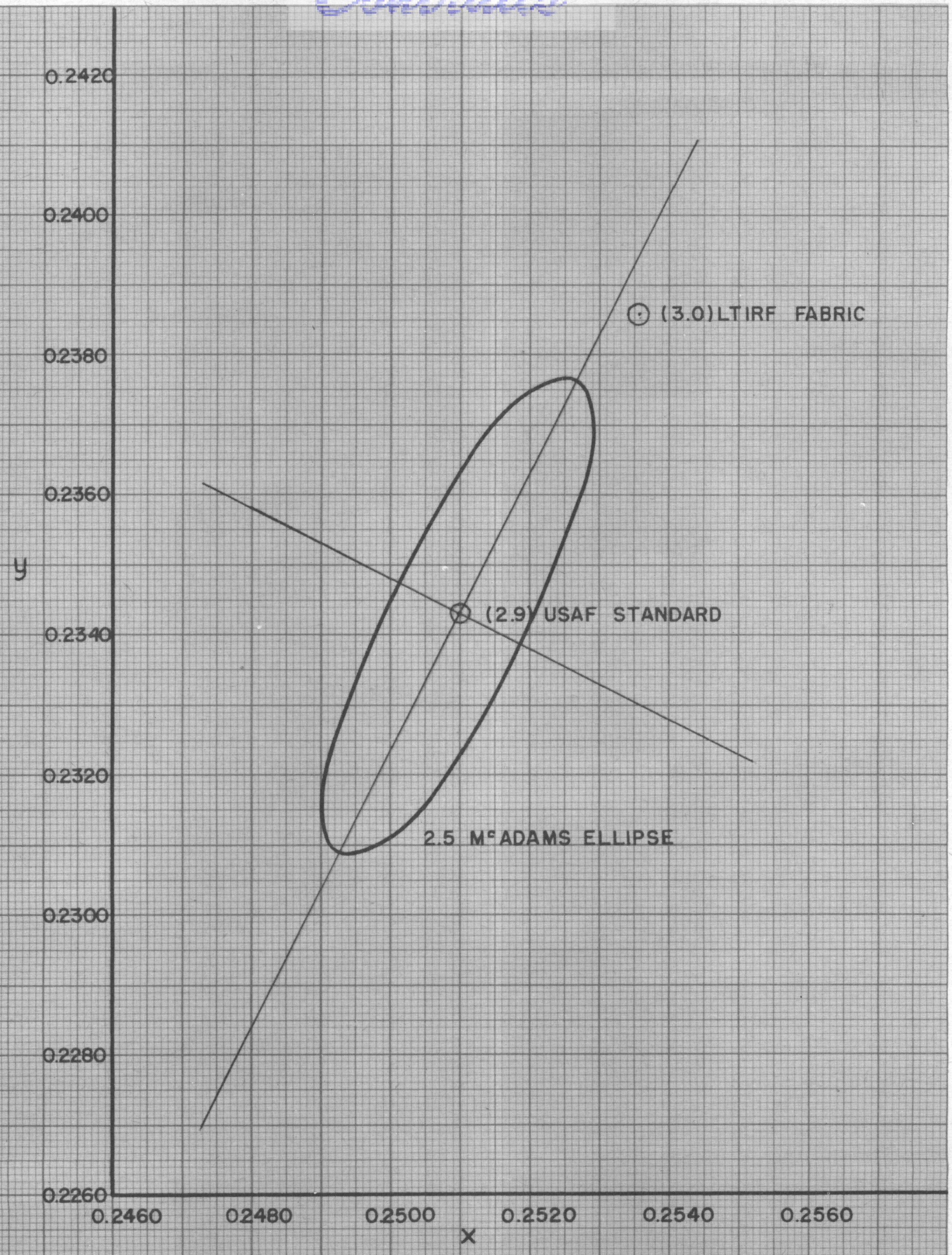

FIGURE 4. CHROMATICITY DIAGRAM - USAF WOOL GABARDINE SHADE BLUE 156

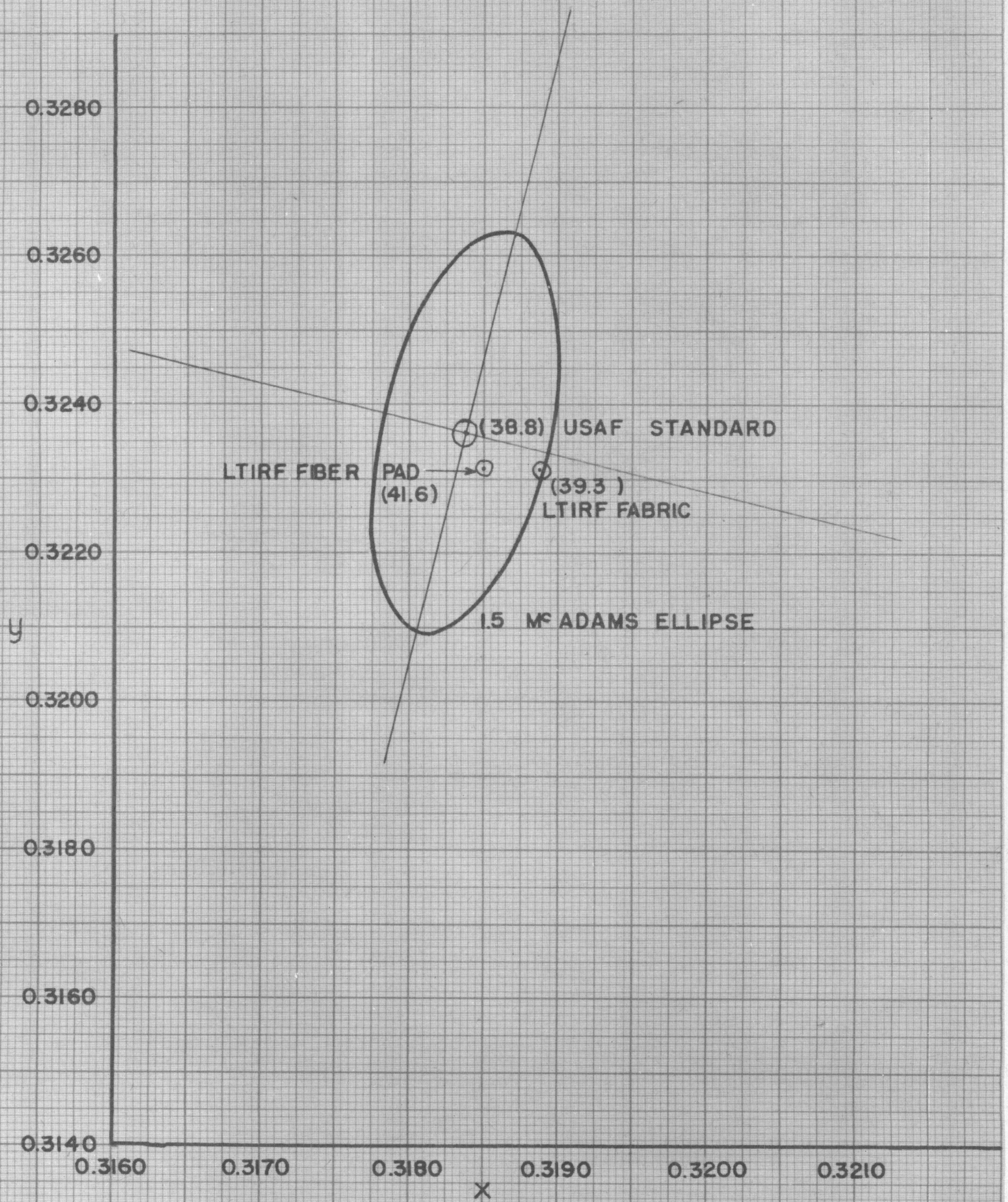

FIGURE 5. CHROMATICITY DIAGRAM - USAF WOOL GABARDINE SHADE GRAY 167

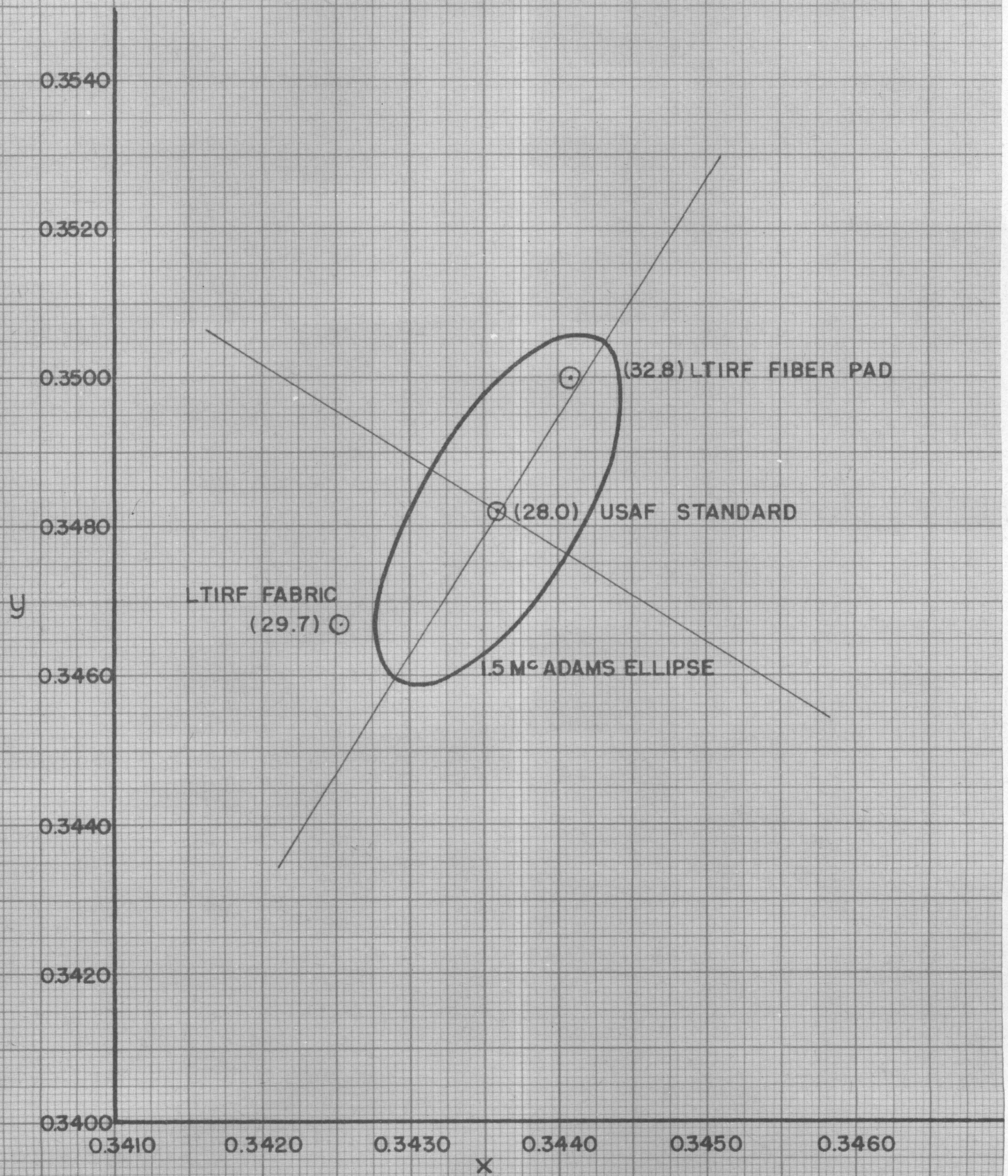

FIGURE 6. CHROMATICITY DIAGRAM - USAF WOOL GABARDINE SHADE TAN 193

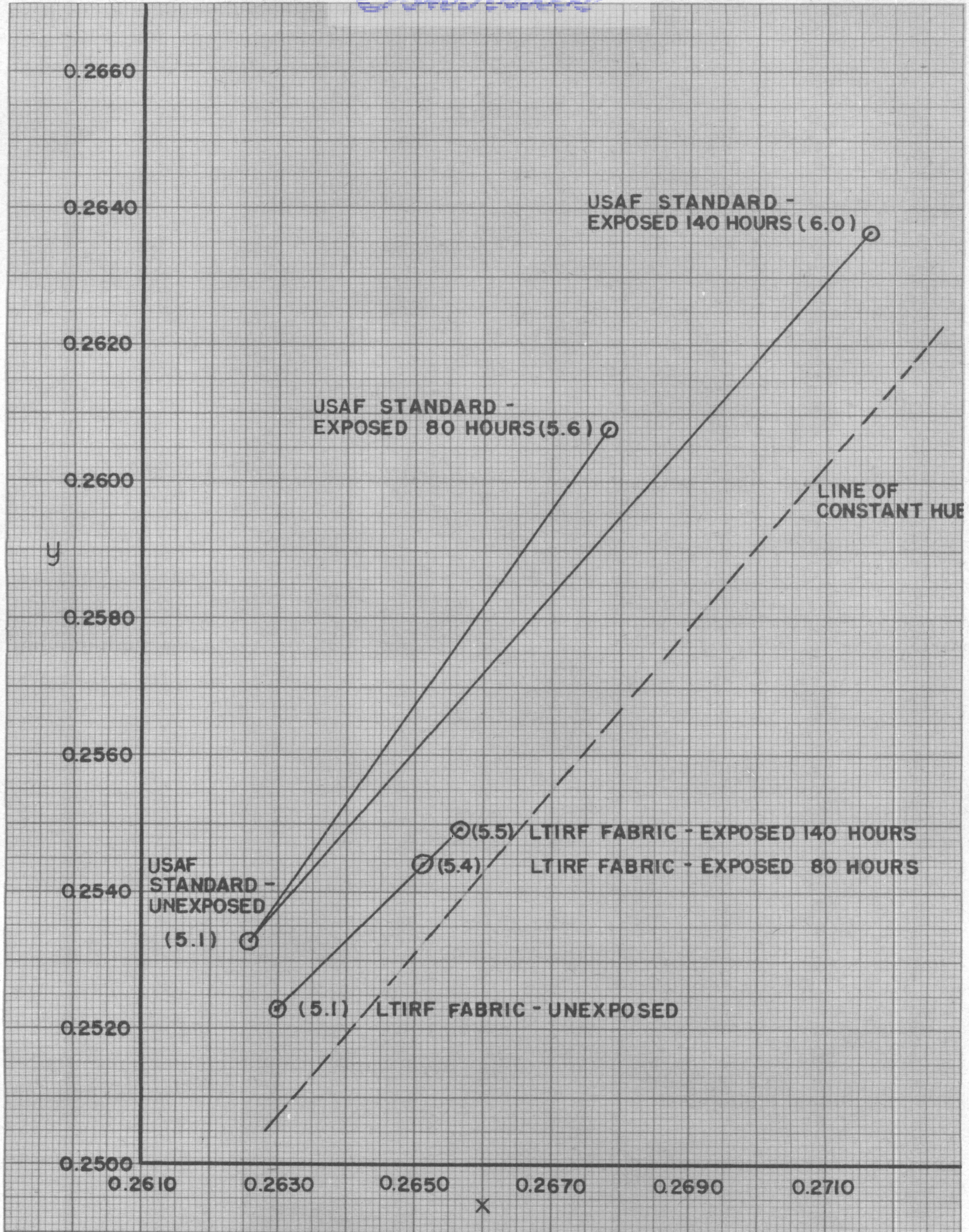

FIGURE 7. CHROMATICITY DIAGRAM OF FADING - USAF WOOL SERGE SHADE BLUE 84

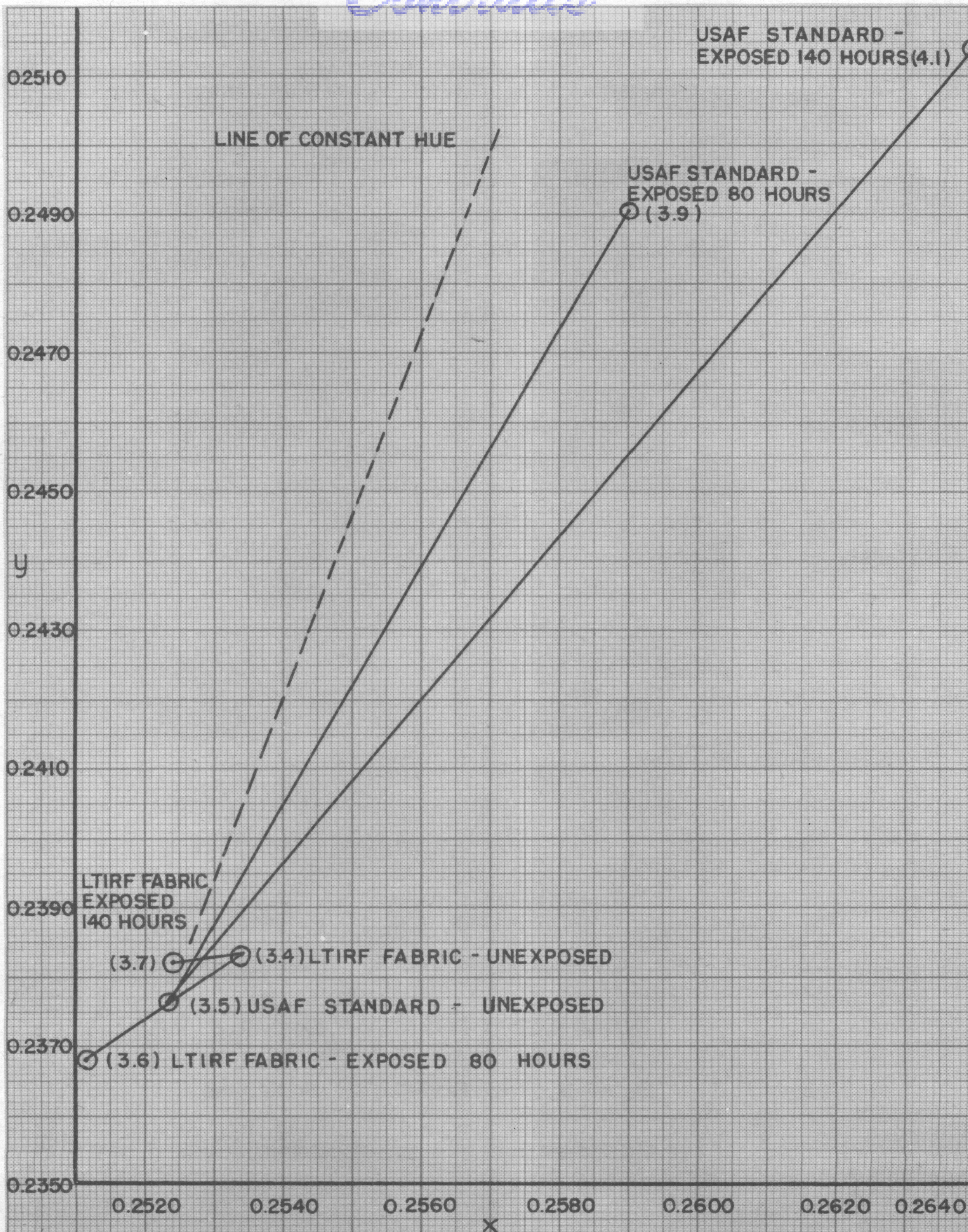

FIGURE 8. CHROMATICITY DIAGRAM OF FADING - USAF WOOL GABARDINE SHADE BLUE 156

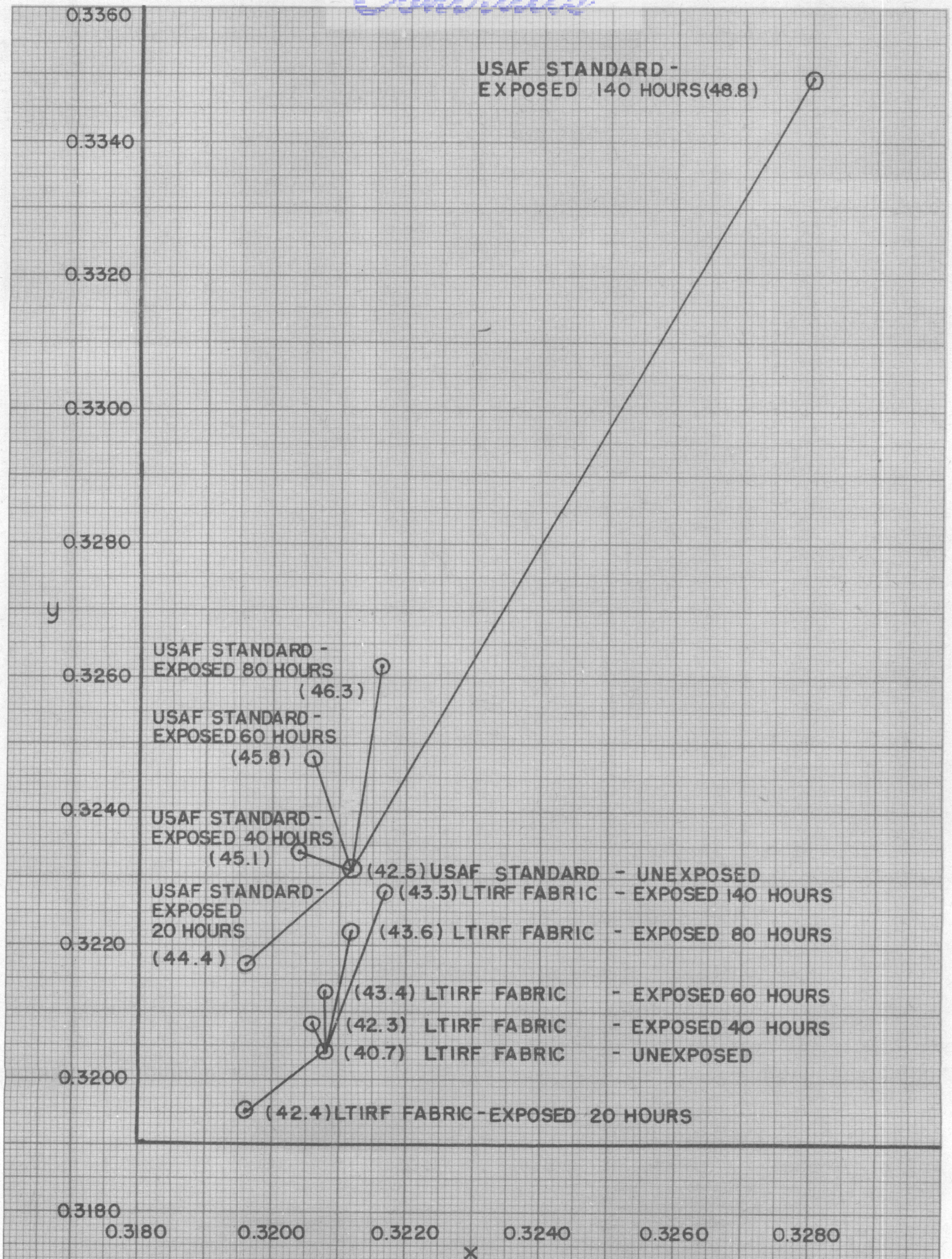

FIGURE 9. CHROMATICITY DIAGRAM OF FADING - USAF WOOL GABARDINE SHADE GRAY 167

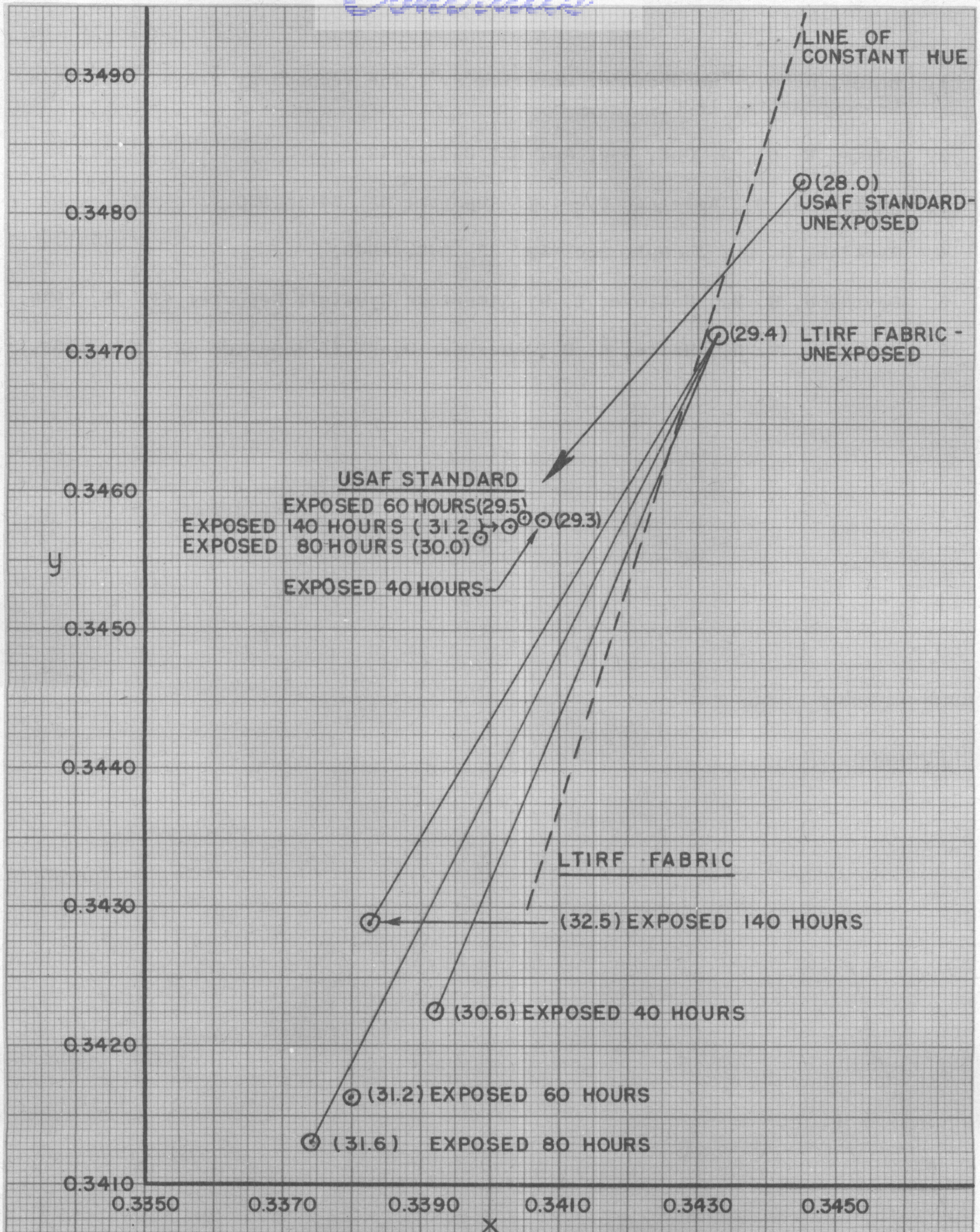

FIGURE 10. CHROMATICITY DIAGRAM OF FADING - USAF WOOL GABARDINE SHADE TAN 193

BIBLIOGRAPHY

1. Hardy, A. C., "Handbook of Colorimetry", The Technology Press, Cambridge, Massachusetts, 1936.

2. Adams, E. Q., J. Opt. Soc. Am. 32, 247, 1942.

3. Nickerson, D., American Dyestuff Reporter 41, 541, 1950.

4. MacAdam, D. L., J. Opt. Soc. Am. 33, 247, 1943.

5. Davidson, H. R., Godlove, I. H., American Dyestuff Reporter 41, 78, 1950.

6. Davidson, H. R., Haire, R. D., American Dyestuff Reporter 42, 253, 1953.

APPENDIX

SPECTROPHOTOMETRY AND COLORIMETRY*

In the work associated with this report wide use has been made of spectrophotometry and colorimetry both in the development of dyestuff formulations and in the control of fabric manufacture. In this appendix, a brief discussion of the theoretical basis for that work is presented.

SPECTROPHOTOMETRY

In its simplest form a spectrophotometer is a device for analyzing the quantity of light a sample reflects or transmits as a function of wavelength relative to a standard (usually MgO). This is accomplished by splitting white light into a spectrum and then illuminating the sample and standard with successive narrow portions of this spectrum. Although the intensity difference which results due to the difference in absorbing power may be evaluated visually, this is quite tedious and subject to many errors. Today it is most frequently done by means of photoelectric cells and the result recorded automatically in a few minutes.

The result of such an operation is a plot of reflectance vs wavelength. By use of special cams it is also possible to plot a function of wavelength which will be proportional to the concentration of dye on the fiber. The most commonly used is the Kubelka-Munk function, i.e. $\frac{1-R^2}{2R}$. By plotting instead the logarithm of this function we obtain a graph in which the curve shape is independent of concentration (1).

In practice, plots such as those described above are used most frequently for shade matching, dye strength measurements, and trouble shooting. Since this report is concerned only with dyestuff formulation and shade matching, it will not be necessary to discuss other applications. In principle the technique is simple and proceeds as follows:

1. The standard to be matched is measured by means of a spectrophotometer with the special "R" cam attached.

2. A careful study of the resulting curve is made with the intention of selecting dyestuffs which will, in combination, closely approximate the standard curve.

* This discussion is based in part on a paper presented to the Textile Federation of Canada by Roland E. Derby, Jr.

3. A "match" is then constructed by combining graphical solutions of the appropriate equations with prepared curves of the selected dyes at known concentrations. If such curves are not available, they must be constructed from experimental dyeings. This "curve" or physical match is of utmost importance in assuring trouble-free matching problems which may occur at a later date.

4. If a combination can be found which possesses the desired properties plus matching the curve, the problem is solved.

In a case where one desired to optimize all properties of the formulation, compromises must be tolerated; however, a match under two or more illuminants is desirable. It should be emphasized that this is a compromise situation and at the earliest possible stage the old standard should be discarded and the new formulation established as the standard.

In the case of matches made by blending primaries of proper shade, a different procedure must be employed. The reason for this is that the laws governing blends are different from those affecting solid shades. Although certain semi-empirical additive relations are known which are of utility in such problems, their use is not as clear cut as the preceding solid shade problem.

The general procedure involved in initial formulation is as follows:

1. On the basis of colorimetry, one selects certain possible primaries.

2. A trial calculation is then made using these primaries by means of the Stearns-Noechel additive function (2).

3. The agreement with curve shape match is then examined and necessary changes in the dyestuff formulations of the primaries or the blend are undertaken.

4. In selecting the primaries it is desirable to employ the minimum number compatible with attaining the desired "heatheriness".

5. The primaries should be so located on a chromaticity diagram that the standard is quite centrally located with respect to the primaries. In other words, the standard blend should fall roughly at the geometric center of the figure formed by joining all the primaries by straight lines in such a manner that each primary is joined directly only to adjacent primaries.

In order to understand better the references to chromaticity, color coordinates, etc., made above, it is desirable to discuss briefly the physical concepts involved and at the same time to include a discussion of color tolerance specification.

COLORIMETRY

It has been demonstrated by thousands of experiments that it is possible to represent adequately the color of a textile material in a given illuminant by three numbers. These numbers are called tristimulus values and are designated X, Y, Z. For an authoritative account of the historical development one should consult "The Science of Color" by The Committee on Colorimetry of The Optical Society of America.

In order to determine the tristimulus values of a given object color, the following data are necessary:

1. The reflectance (or transmittance) of the material as a function of wavelength throughout the visible spectrum (400-700 millimicrons).

2. The energy distribution of the illuminant being considered (i.e., daylight, tungsten light, etc.) presented as a function of wavelength over the 400-700 millimicron range.

3. The basic color-matching characteristics of an average observer over the visible range of wavelengths.

The basic data for Item 1 may be readily obtained by means of a spectrophotometer. The necessary information regarding the energy distribution, Item 2, has been experimentally determined by spectroradiometry for average daylight (Illuminant C) and tungsten light (illuminant A). These results have been made definitive by the C.I.E. (International Commission on Illumination).

Lastly, the ability of the average observer to discriminate between near color matches is represented by fundamental data determined by careful experiments. The results are represented by three curves called by tristimulus functions $\bar{x}$, $\bar{y}$, $\bar{z}$. The significance of these curves is perhaps best understood by noting that at any given wavelength the value of the function represents the amount of a given primary necessary to match a spectrum color of that wavelength. In other words, if one additively mixed the CIE primaries in the proportions indicated at a particular wavelength, the average observer would consider the mixture to be a match for the spectrum color. It should be noted that the selection of primaries is not unique, since the resulting specification based on one set can be transformed into that based on another mathematically, provided the relationship of one set of primaries to the other is known. It is also important to realize that the data specify an average of several observers and thus can be considered more representative than a single observer.

In order to calculate the tristimulus values X, Y, Z, one must obtain the product of these functions over the visible region. Such a process may be performed by integration, where the tristimulus values are:

$$X = \int_{400}^{700} E(\lambda)R(\lambda)\bar{x}(\lambda)\,d\lambda$$

$$Y = \int_{400}^{700} E(\lambda)R(\lambda)\bar{y}(\lambda)\,d\lambda$$

$$Z = \int_{400}^{700} E(\lambda)R(\lambda)\bar{z}(\lambda)\,d\lambda$$

Since the products $(E \cdot \bar{x})$, $(E \cdot \bar{y})$, and $(E \cdot \bar{z})$ are constant, it is only necessary to evaluate R (the reflectance). As previously noted, this information is readily obtained by spectrophotometry. In this project all integrations were performed mechanically, the appropriate integrals being evaluated as rapidly as the recording Spectrophotometer determines the reflectance of the sample, i.e., approximately 2-1/2 minutes per sample.

In most cases it is more convenient to plot "color coordinates" obtained from the following relations:

$$x = \frac{X}{X + Y + Z}$$

$$y = \frac{Y}{X + Y + Z}$$

note: $z = 1 - (x + y)$

The primaries of the CIE system were originally chosen so that Y (in appropriate units) indicates the photometric reflectance (designated as the apparent luminous reflectance). Thus it is usually considered that x, y indicated chromaticity, namely hue and saturation of a given sample, while Y indicates its lightness. In conventional plotting techniques, x, y, and Y are plotted in rectangular coordinates.

In this brief discussion it is impossible to justify the technique or the data. Perhaps the best justification is that in over twenty years thousands of color calculations based on this system have revealed few significant deviations between visual experience and properly measured results.

GRAPHICAL REPRESENTATION OF COLOR

The conventional method of plotting color was outlined in the previous section, that is, in a rectangular coordinate system. Such a plot is called a "chromaticity diagram".

A most important development in the utility of the colorimetric method occurred when a plot was devised wherein the standard always appeared at the origin of the coordinate system regardless of the absolute position in color space. All samples are then plotted by their difference from the standard. This procedure was originally implied in a paper by MacAdam (3) and has been recently utilized extensively in publications by H. R. Davidson (4).

It has proven of value considerably beyond the original reasons for its development. Let us consider its role in the generalized problem of color control with reference to a specific standard color.

First, a suitable standard in the form of a swatch of material is agreed upon by those concerned. The colorimetric specification of the material is then determined with great care in the manner previously outlined. It is very important at this stage to assure adequate calibration of the spectrophotometer according to procedures recommended by the National Bureau of Standards. The automatic integrator should also be carefully calibrated at the same time. Several measurements (<u>at least 2</u>) should be made on the standard in order to specify the precision of measurement. If sufficient material is available, the standard is then divided in half, one half becoming a master standard and the other a working standard.

A chart is next prepared as shown in Figure 1. In this figure it will be noted that the standard (S) appears at the origin. Δx and Δy, the differences in color coordinates between the standard and any sample are plotted on the abscissa and ordinate respectively. This procedure tends to minimize the periodic or random instrumental variations (of a minor nature) which may enter into an <u>exact</u> specification of a given sample. The points P_1, P_2, and P_3 refer to particular samples measured and plotted as described. The lightness difference ΔY is shown in parenthesis beside the appropriate point.

In Figure 1 an "acceptability" ellipse has been drawn about the standard. The ratio of the major and minor axis of this ellipse may be readily determined from data published by MacAdam (3). The dimensions of the ellipse in terms of the scale units being used are best determined experimentally; considerable variation being encountered depending on the type of material being considered and the purpose for which it is intended. However, an ellipse which is 2.5 times the unit ellipse specified by the values given by MacAdam has been found to be satisfactory for this project. Lighter shades will require smaller multipliers and darker shades somewhat larger ones.

There are several ways of representing the Δ Y tolerance. For general control work a method of considerable utility is derived from the familiar contour terrain map. In this method a chart is prepared exactly as in Figure 1 with the exception that it is composed solely of a series of concentric ellipses inside the one shown. Each ellipse represents a line of constant Δ Y tolerance. This would be a maximum (corresponding to the top of the hill) when Δ x and Δ y = 0 and zero when the point falls on the edge of the ellipse (the one representing chromaticity tolerance). This is prepared on transparent paper, and it is merely necessary to place this over the chart shown in Figure 1 and note whether the point has too great a Δ Y for the particular chromaticity involved. For example, see point P_3 in the diagram where the value (+ 10) indicates it is too light. Point P_2, while adequate for depth, is "off shade", falling outside the ellipse. It should be pointed out that in certain cases the lightness tolerance on the heavy side will not be equal numerically to that on the light side (as indeed it should not be due to the Weber-Fechner relation, wherein $\Delta Y/_Y$ = constant). This fact necessitates having two transparent charts, one for dark samples and another for light. It is also possible to present contours in different colors or even more simply a different color may be used in printing in the value assigned to the particular contour.

In Figure 1 it will be noted that designations such as redder, bluer, etc., have been shown by arrows. This is not a general scheme, for the particular designation depends on the shade being considered. However, such a chart can be easily prepared, for example, by reference to a book of Munsell colors and their basic colorimetric data (5).

A special plot devised by Davidson & Hanlon (6) is sometimes useful. In this case a new chart is prepared in which the x axis consists of units obtained by dividing the distance to any point (say P_2 in Figure 1) by the distance to the edge of the tolerance ellipse along the line joining the standard to the piece (i.e., P_2). On this scale (1) represents a tolerance when there is no lightness difference (Δ Y). The Δ Y value for the sample is then plotted on the y axis. Under these conditions the Δ Y scale may be adjusted so that a circle results in which 1 unit (along the y axis) at zero chromaticity is equivalent to 1 unit (along the x axis) at zero lightness difference. Such a plot has the decided advantage that linear distances are directly equal to color differences. However, a somewhat empirical calibration must be made for each shade. This technique should prove most useful in cases where conditions are fixed and a large number of color differences are to be evaluated.

By plotting samples at various stages of production, it is possible to establish the "average" change which a piece undergoes in finishing. Thus, it is possible to predict where (on the chromaticity plot) future samples must fall in order that on the average a piece matching the standard will result.

FADING TEST EVALUATION

Since a considerable number of lightfastness test results have been published during the course of this project, a brief discussion of the procedure used would seem to be in order. The following outline indicates the procedure:

1. The unexposed area and exposed area are measured (two measurements on each area).

2. The color difference between areas is determined by a suitable formula. At present this is the Adams-Nickerson formula; complete details for its use being given in the Dyestuff Reporter (7).

3. The chromaticity and lightness of the two areas are plotted on a chart in the manner described earlier. These points are connected by a line.

4. A Munsell Constant Hue Line is drawn on the chart (5).

5. Steps 1 through 3 are carried out for the standard and all samples.

In order to clarify certain points regarding these charts, the following points should be noted:

1. In evaluating light tests a sharp line exists between faded and unfaded sections. It has been recognized for many years that this causes difficulty in attempting to employ color difference formulae which were developed for general conditions of sample comparison, i.e., two swatches. It is therefore important to consider carefully the data and the nature of the variations in using equations of the Adam's type.

2. Generally, if a sample fades parallel to a Munsell Constant Hue Line over a short distance, it will be more desirable than one which does not (if both show "equal" color differences). At any rate the degree of non-parallelism should not be greater than the standard.

3. If a change of formulation should be made, it is readily apparent by a drastic change of slope.

4. Due to the fact that there is a tolerance around the standard (i.e., the ellipse shown), it is frequently possible that a dyeing may exhibit inferior or better fastness than the standard, even though both are dyed with the same formulation. In other words, a piece may fall within the ellipse and be acceptable. However, on fading, this piece may fade off tone due to the slight unbalance of colors. That is, it may move more obliquely to the Munsell Constant Hue Line or a greater distance. This is the danger of adding so-called "shading colors".

The method outlined is useful as a quality control procedure. For example, if it specified that the sample must show less fading than a standard, then routine sampling of current production may be tested by the criteria of a smaller color difference and equal or better parallelism.

If standards and tolerances were set up on this basis, particularly in referee cases, most discussion and argument could be avoided. That this has been done only rarely is a matter of some concern.

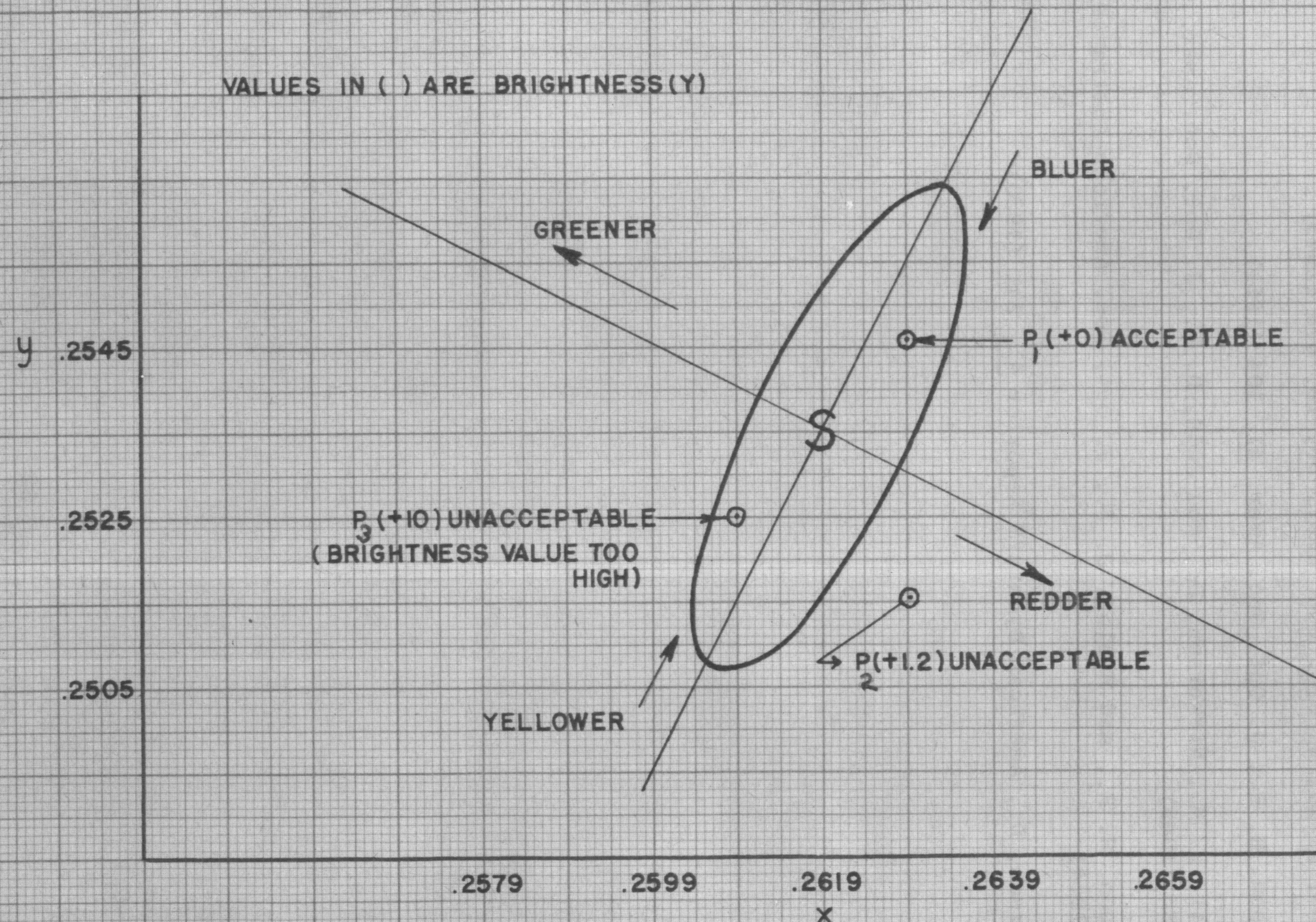

FIGURE I. DEFINITION OF CHROMATICITY DIAGRAM

REFERENCES

1. Derby, R. E. Jr., American Dyestuff Reporter 41, 550 (1952)
2. Stearns, E. I., and Noechel, F., American Dyestuff Reporter 33, 177 (1944)
3. MacAdam, D. L., J. Opt. Soc. Amer. 33, 18, 675 (1943)
4. Davidson, H. R., J. Opt. Soc. Amer. 41, 104 (1951)
5. Newhall, S. M., Nickerson, D., and Judd, D. B., J. Opt. Soc. Amer. 33, 385 (1943)
6. Davidson, H. R., and Hanlon, J. J., J. Opt. Soc. American Abstracts 44, 350 (1954)
7. Nickerson, D., American Dyestuff Reporter 41, 541 (1950)