1942

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Recommended Citation

Donohue, Ruth Olive, "The chemical resistance of polyhexamethylene adipamide " (1942). *Retrospective Theses and Dissertations*. 13519.
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THE CHEMICAL RESISTANCE OF POLYHEXAMETHYLENE ADIPAMIDE

by

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A Thesis Submitted to the Graduate Faculty
for the Degree of

DOCTOR OF PHILOSOPHY

Major Subject: Textile Chemistry

Approved:

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In Charge of Major Work

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Dean of Graduate College

Iowa State College
1942

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INTRODUCTION

This investigation was undertaken to obtain quantitative information about the chemical resistance of fibrous polyhexamethylene adipamide.

REVIEW OF LITERATURE

Production of Nylons

The production of a nylon which may be used as a textile was a development of ten years' investigation of high polymeric reactions by W. H. Carothers and his coworkers for E. I. du Pont de Nemours and Company (79, 80, 81, 152).

Carothers and Hill (84) first succeeded in preparing filaments from linear superpolyesters (molecular weight, 10,000 to 12,000) made by heating α-polyesters (molecular weight, 800 to 5000) in a molecular still (75, 83). Fischer and others (102, 105, 170) had previously prepared polyamides through the condensation of α-amino acids or their derivatives, but these products had shown no fibrous characteristics. Carothers and Hill (82, 86) next reported that a superpolyamide from ε-aminocaproic acid was too insoluble and too infusible for spinning but that a mixed polyester-polyamide polymer yielded a considerably stronger filament (85, U. S. Patents 2,071,250 and 2,224,037). Although strong and pliable filaments were also obtained from ω-superpolyanhydrides and superpolyacetals, none of these filaments was found to have suitable textile properties (123, 124, U. S. Patent 2,071,252).

Carothers and his coworkers continued to investigate the production of polyamides. British Patent 461,236 describes the condensation of amino acids (of the formula $NH_2(CH_2)_nCOOH$ where n is 5 to 10, or 12, 16, or 21) at high temperature in an inert solvent and an atmosphere of nitrogen for the production of a spinnable filament (61). On February 28, 1935, the superpolymer from hexamethylene diamine (18, 71, 111, 146, British Patents 490,922 and 494,236, U. S. Patent 2,181,140) and adipic acid (92, 107, 160, 193, 212, U. S. Patents 1,960,211 and 2,191,786) was synthesized. Although polymers from other diamines and dibasic acids and interpolymers from diamines, dibasic acids, and amino acids (British Patents 535,262; 540,134; 540,135) were investigated, polyhexamethylene adipamide was found to show the best textile properties at the lowest cost of production.

The production of superpolyamides through the condensation of a primary or secondary diamine with a dicarboxylic acid or its derivative is described in British Patent 461,237. The process consists in the refluxing of equimolar quantities of a dibasic acid of more than two carbon atoms with a diamine of more than three carbon atoms at 120 to 190°C. in an atmosphere of nitrogen. The salt formed (British Patent 474,999) is recovered and heated in an autoclave at 200 to 290°C. with an agent for stabilizing the viscosity of the melt (British Patents 495,790 and 535,335, U. S. Patents 2,174,527 and 2,264,293) until the intrinsic viscosity nears 0.4 (150, 173, 211). The superpolymer is then extruded as a ribbon, cut into chips, and thoroughly mixed; these chips are heated above 263°C. in an inert atmosphere, a delustrant is added (67, British Patents 504,714 and 543,125, French Patent 827,798, U. S. Patents 2,205,722; 2,265,127; 2,278,878), the superpolyamide is extruded as a filament, and drawn cold to four times its original length (6, 9, 14, 26, 29, 30, 31, 32, 41, 65, 70, 89, 145, 180, 186, 213, 214, 240, British Patents 491,111; 501,197; 501,527; 504,344; 506,125; 520,263; 533,304; 533,306; 533,307; 535,139; 535,141, U. S. Patents 2,130,523 and 2,130,947).

After October 27, 1938, when Dr. C. M. A. Stine announced the industrial production of the textile fiber, Fiber 66, from "coal, air and water" (43, 97, 182), the preparation, properties, and uses of this fiber received much advertisement in the literature (11, 12, 13, 17, 20, 22, 36, 38, 44, 51, 54, 66, 69, 76, 91, 95, 121, 131, 135, 136, 137, 144, 147, 153, 154, 157, 167, 171, 175, 176, 178, 179, 183, 190, 191, 195, 197, 200, 206, 216, 217, 218, 224, 225, 227, 228, 239). The term nylon was coined as a generic name for the class of superpolyamides made by the condensation of a primary or secondary diamine and a dicarboxylic acid or its derivative (4, 7, 24).

Further patents have dealt with the production of filaments from polymers related to nylon. In an attempt to obtain filaments of greater flexibility, better thermal properties, and different behavior toward some types of dyes, polymers containing sulfur or other hetero-atoms in the chain have been prepared.

Polyamides prepared by the condensation of a diamine containing one or more hetero-atoms (oxygen, selenium, sulfur, or tellurium) with a dicarboxylic or carbothiolic acid have been found suitable for bristles, safety-glass interlayers, and coating compositions although too soluble in most common solvents for use as a textile (8, 92, British Patents 487,734 and 538,120, U. S. Patents 2,158,064; 2,176,074; 2,191,556; 2,223,916). Long-chain polythioureas, made by the condensation of carbon disulfide and hexamethylene diamine in 95 per cent ethanol (British Patents 524,795 and 534,699), and linear polycarbothionamides, produced by heating hydrogen sulfide with a dinitrile of more than five carbon atoms and an aliphatic diamine of at least two carbon atoms (British Patents 528,437 and 535,934, U. S. Patent 2,276,160), have also been reported to be more suitable for coating compositions. However, an oriented fiber has been produced by cold drawing to 173 per cent a filament of a polyamide-polysulfonamide polymer made from hexamethylene diamine and the diacid prepared by hydrolyzing trimethylenedisulfonyl-bisaminocaproic nitrile (215, British Patent 523,506). Polyamides derived from meta aromatic amines and dicarboxylic acids have also been prepared in an effort to obtain fibers having increased resistance to insects (British Patent 525,516). A filament resembling wool has been produced by crimping the fibers of a nylon in the presence of sulfite (40, 50, 119, 205, 237, British Patents 514,821; 530,833; 535,140; 535,379, French Patent 833,755, U. S.

Patents 2,157,117; 2,157,118; 2,174,878; 2,197,896). British Patent 544,820 describes the crimping of nylon with zinc chloride, a metal thiocyanate, or a dihydroxy or trihydroxy benzene and steam.

Sheets, films, and molded articles have been made from:

(a) polyamides derived from polyamines of three or more reactive amino groups and polybasic acids of three or more carboxylic groups (U. S. Patent 2,149,273);

(b) polyamides prepared from unsaturated aliphatic diamines and dicarboxylic acids (British Patent 526,942, U. S. Patents 2,174,619 and 2,238,682);

(c) polyamide-rubber compositions containing 25 per cent of polyamide (British Patent 535,070 and U. S. Patent 2,249,686);

(d) plasticized compositions prepared by mixing an alkyl arylsulfonamide, a cyclic ketone like camphor, a phenol, a halogenated hydrocarbon such as a polychlorinated biphenyl, or a sulfonamide-formaldehyde product with a polyamide (British Patents 533,967; 536,130; 542,838, U. S. Patents 2,214,397; 2,214,402; 2,214,405; 2,216,835; 2,243,662; 2,244,184; 2,276,437);

(e) polyamides prepared from cyclic amides of more than six annular atoms (British Patent 538,619, U. S. Patents 2,241,321; 2,241,322; 2,251,519; 2,276,164; 2,277,152);

and from (f) a polymerized amide or imide prepared by the action of heat or x-rays on a N-vinyl tertiary amide or imide of an aliphatic carboxylic acid (British Patents 543,426 and 543,566).

While seeking a substitute for commercial nylon, Hosino (130) prepared polycyclohexane-1,4-adipamide and poly-1,4-cyclohexylenethioureide. He obtained a number of substitutes for nylon by heating varying proportions of 4-aminocyclohexyldithiocarbamic acid with hexamethylene adipamide in an atmosphere of hydrogen. Mokudai and Oda (161) have reported that they obtained a fiber-forming polymer when they converted cylcohexanone oxime (British Patent 535,421) into the "iso-oxime" by the Beckmann reaction and heated the product in a sealed tube at 300°C.

Properties of Commercial Nylon

Fuller (109) has observed a period of 17.0Å in the x-ray diagram of an oriented fiber of polyhexamethylene adipamide; that calculated on the basis of a zigzag plane is 17.3Å (151). Sakurada and Hizawa (184) have obtained a value of 17.3Å for this period and a value of 3.5Å for the length of the amide group; from this they computed the unit of the chain as fourteen atoms, twelve carbon atoms and two nitrogen atoms corresponding to hexamethylene adipamide. More recently Baker and Fuller (58) have obtained a value of 17.4Å for the repeating unit of this fiber. By examination with x-rays and determination of Young's modulus (174), Fuller, Baker, and Pape (110) have studied the effect of heat on solutions of polyhexamethylene adipamide and polyhexamethylene sebacamide in m-cresol. Their data support the idea that segments of long-chain molecules may exist as crystalline, mesomorphic, or amorphous regions in a solid polymer (148, 149).

By determination of the viscosity of dilute solutions of commercial nylon in m-cresol, Staudinger (199) has obtained 12,500 as the mean molar weight of this polymer; this value corresponds to 920 units per chain (96, 104, 129).

Harris and Sookne (122) have studied the electrophoretic behavior of both unoriented and oriented fibers of polyhexamethylene adipamide and have observed that both fibers exhibit a reversal of charge, the unoriented fiber at pH 3.9 and the oriented fiber at pH 2.7.

Some other physical properties of the nylon fiber determined and made public by the Nylon Division of E. I. du Pont de Nemours and Company (34, 39, 46, 50, 140, 166, 185) are:

A. Electrical properties	
1. Breakdown strength	1210 volts per mil.
2. Dielectric constant, 20°C.	5 at 1000 cycles
3. Power factor, 20°C.	2 to 3 per cent at 1000 cycles
4. Volume resistivity at	
a. 18 per cent R. H.	4×10^{14} ohms per cm.
b. 100 per cent R. H.	5×10^{9} ohms per cm. (39)
B. Tensile properties	
1. Dry strength	5 g. per denier
2. Dry elongation	20 per cent, but varies widely
3. Wet strength	4.4 g. per denier
4. Wet elongation	30 per cent, but varies widely
5. Dry loop strength	95 per cent of tensile strength
6. Elastic recovery when stretched to:	
a. 8 per cent	100 per cent
b. 16 per cent	91 per cent
7. Hardness	380 kg. per sq. mm. (185)
8. Modulus of elasticity	505 kg. per sq. mm. (140)

C. Thermal properties

1. Mean heat capacity, 20 to 265°C.	0.555 cal. per g. per °C.
2. Heat of fusion	22 cal. per g.

Staudinger (199) has compared the tensile strength and degree of polymerization of nylon and some other textile fibers.

Fiber	Degree of polymerization	Yarn number	Breaking strength dry	Elongation
		denier	gram per denier	percentage
Rayon				
Cellulose-nitrate	170	6.5	1.75	17.5
Cuprammonium	560	3.9	1.97	26.0
Viscose	290	1.4	2.35	16.0
Egyptian cotton	3000	1.85	3.6	13.1
Ramie	3000	4.1	6.7	4.1
Lanital		3.3	0.9	84.2
Silk		1.25	5.8	21.5
Wool		5.5	1.5	41.4
Polyvinylchloride	ca 2000	4.1	1.4	24.0
Nylon		3.2	4.25	16.2

Ashmore (55) has compared woven fabrics of cotton, nylon, silk, and viscose rayon designed for parachutes.

Determination	Cotton	Nylon	Silk	Viscose Rayon
Weight, <u>ounces per square yard</u>	2.05	1.8	1.6	1.8
Breaking strength				
Warp, <u>pounds per inch</u>	38	60	40	50
Filling, <u>pounds per inch</u>	38	60	40	50
Tearing strength				
Warp, <u>pound</u>	2.5	4.0	4.0	2.0
Filling, <u>pound</u>	2.5	4.0	4.0	2.0
Weave	2/1 rib	2/1 rib	2/1 rib	plain
Air porosity, <u>ft.^{3} of air per ft.^{2} of fabric in 0.5 in. of water</u>	80-140	80-140	80-140	80-160

Cleary (90) has determined the strength-weight ratios of cotton, linen and nylon webbings for the purpose of comparing their efficiency in parachutes. The strength-weight ratios, 27.8 for cotton, 30.9 for linen, and 41.6 for nylon, he described as in accordance with the inherent properties of these fibers.

Nylon has been reported as resistant to moths and mildew but as not water-repellent unless processed with formaldehyde or <u>Zelan</u> (35, 39, 50). When suspended vertically over a gas burner and ignited, nylon was observed to soften, melt, and burn, but not to keep burning (56).

Most solvents have been reported to have no effect on nylon at room temperature; phenol, m-cresol, xylenol, formic acid, and a mixture of methanol and chloroform, however, dissolve nylon at room temperature (50, 77, 166, British Patents 542,444; 543,062; 543,063). At higher temperatures a few other compounds have been described as solvents for nylon (166).

Methods for identifying fibers of polyhexamethylene adipamide include ignition, microscopic examination, tests for solubility in acetone, hydrochloric acid, and phenol, and staining reactions with Brilliant Blue 6BA, National Soluble Blue 2B Extra, and Colotex B (21, 33, 45, 53, 56, 94, 159, 196, 229). Procedures for the quantitative estimation of nylon in textile mixtures have been developed by Freedman and Nechamkin (106) and Gerber and Lathrop (113, 114).

Descriptions of the chemical properties of polyhexamethylene adipamide have been more qualitative than quantitative. This fiber has been reported as decomposed with boiling 5 per cent hydrochloric acid (166). Cold concentrated inorganic acids have been reported to hydrolyze the fiber, whereas cold dilute solutions (less than 5 per cent) have had no appreciable effect (201). Stott (201) has reported that the fiber dissolves in concentrations of formic acid greater than 10 per cent and that acetic acid has neither dissolving nor swelling action on the fiber.

von Bergen (62) has noticed that filaments of nylon boiled for one hour in 4 per cent sulfuric acid increase 3.2 per cent in diameter when measured in glycerol. He has also compared the swelling of nylon in glycerol, water, sodium hydroxide, and in a dye bath (62, 63).

Nylon	Glycerol	Water	Sodium hydroxide 10 per cent	Acid dye, 4 per cent sulfuric acid, 10 per cent $Na_2SO_4.10H_2O$
Number of fibers	100	100	100	100
Percentage of fibers of				
a. 10 to 20 micron	99	95	89	78
b. more than 20 micron	3	5	11	22
Mean diameter, micron	18.53	18.58	18.55	19.12
Deviation	1.00	1.03	1.35	1.30

Filaments of nylon or silk carbonized with 4 per cent sulfuric acid for one hour and then dried at 105°C. for one hour have been shown to decrease in tensile strength; the mean loss in ten determinations was 30.3 per cent for nylon and 74.8 per cent for silk while the elongation decreased 21.5 per cent in the case of nylon and 75.5 per cent in the case of silk (62).

Stott (201, 202, 203), Wakelin (223), and White (232) have investigated the dyeing of nylon with many types of dyes including Acetamine, acid, basic, Bernacyl, Celanthrene, chrome, developed, direct, Soledon, sulfur, and vat colors. Stott and others (68, 78, 155, 204, 233) have reported that cellulose-acetate dyes are best adapted for nylon but that acid, basic, and chrome dyes may be used, although microscopic examination of this fiber dyed with an acid color has revealed that an acid dye does not penetrate the inner part of the filament (202). Each acid dye has a saturation point beyond which it is not fixed by the fibrous nylon (64). When a nylon fiber was dyed in "a mixture of acid dyes", cross-sectioned, and examined microscopically, it was found that the acid dyes had penetrated so that there was an outer brown ring, an inner green ring, and an innermost yellow ring (181). White (232) has shown that an acid dye possesses a fastness to washing on nylon superior to that on silk. Oil-soluble colors which dye cellulose-acetate rayon orange will dye nylon red; those red or blue on acetate rayon become blue or green, respectively, on nylon (181). Certain basic dyes which show only feeble fluorescence on other fibers have been shown highly fluorescent on nylon (232). Nylon dyed with Bernacyl Colors has shown inferior fastness to heat, light, sweat, and washing (64). Nylon has been reported to have little affinity for the majority of direct, sulfur, and vat dyes (64, 68) but to be dyed readily with certain azo dyes (British Patents 539,195 and 543,198). Meunier (156) has studied the printing of acetate, acid, chrome, direct, and vat dyes on nylon with thickeners containing 50 per cent British gum and has noted that the fastness of acid, anthraquinone, and direct colors can be increased by the addition of urea or glycolic acid to the printing paste (British Patent 534,085). Tsimēhc (213) has investigated the dyeing of polyamides stabilized with a free terminal acid group and has shown that this product has greater affinity for acid dyes; when the molecule is stabilized with a free basic terminal group, the affinity for direct dyes is greater.

The dyeing of nylon with acetate dyes has been postulated to take place through the formation of a solid solution (181). Dyeing with acid dye from acid solution may be considered a hydrogen bonding (60, 116, 181).

The stripping of dyes from nylon with 1:3 ammonium hydroxide at 40°C., solutions of sodium or zinc hyposulfite at 40 to 50°C., or boiling solutions of titanium salts has been shown to have little effect on the fiber (77, 231). Hydrogen peroxide, 3 per cent for two hours or 0.2 per cent for sixteen hours, has been shown to be without effect on nylon and not even to bleach it (166).

Uses of Commercial Nylon

One of the most general uses of the fiber of polyhexamethylene adipamide has been in the production of hosiery (1, 5, 37, 49, 134, 139, 164, 189, 226, 230). The fiber as prepared for knitting contains a size consisting of 6 per cent polyvinyl alcohol, 2 per cent boric acid, and 92 per cent water (188, 219, 220); this size is removed with an alkali alkyl sulfate, pH 12, after the "pre-boarding" process (163). The temperature and humidity of the knitting room is carefully controlled at 80°F. and 55 per cent R. H. lest a variation of more than 2 in. in the width of a stocking occur (27, 142, 198). The "pre-boarding" or "setting" process is accomplished by placing the hosiery on forms for treatment with (a) boiling water for ten minutes, (b) steam at atmospheric pressure in a drying chamber, or (c) live steam at a pressure of 5 to 15 lb. for 2.5 to five minutes (52, 143, 187, British Patents 524,953 and 530,833, U. S. Patent 2,226,529). The hosiery is then scoured, dyed, and finished with a resin to diminish snagging (162, 233).

Physiological reaction toward polyhexamethylene adipamide has not been definitely proven. In several cases patients who suffered from a dermatitis after wearing nylon hosiery were proved allergic to the abietic ester applied as a finish (99, 234); in the one case in which the dermatitis was traced to the fiber, the patient was subject to other allergies (115).

Other uses for nylon are in the production of: bearings (120); brushes (10, 19, 23); bushings (132); crease-proof pile fabrics (British Patent 520,323, U. S. Patent 2,238,098); cross hairs for telescopes (132); coating for fabric, leather and paper (120); filter cloth (132); fishing line (133); gloves (72); insulation (47, 132, British Patent 534,159, French Patent 838,776, U. S. Patent 2,260,024); men's and women's clothing (28); non-crushable velvets (120, British Patent 520,323); paper money and government bonds (120); parachutes (55, 158); powder bags (132); rope and string (132); shoes (120); surgical dressings and sutures (28); and sewing thread (25, 42, 48, 120, British Patent 519,038).

Fibers of polyhexamethylene adipamide are produced on a commercial scale by E. I. du Pont de Nemours and Company in two plants in the United States, by Courtaulds Combine in one plant each in Canada and England and by Rhodiaceta in Italy (15, 73, 118, 169). In Germany the polymer is called Igamide and the fiber, Perlon; other nylons than polyhexamethylene adipamide are also manufactured in Germany for use as adhesives and lacquers (16, 126, 127).

ANALYSIS OF THE FABRIC

Materials

1. Acetanilide. C. P. Eastman Kodak Company.

2. Acetic acid. Glacial, 99.5 per cent. C. P. General Chemical Company.

3. Alizarin Blue S. National medicinal product. National Aniline and Chemical Company.

One gram of Alizarin Blue S was dissolved in 100 ml. of distilled water.

4. Ammonium hydroxide. C. P. Sp. gr. 0.90. General Chemical Company.

5. Ammonium salt of nitrosophenylhydroxylamine. Cupferron. C. P. General Chemical Company.

Four grams of cupferron were dissolved in 100 ml. of distilled water.

6. Ammonium sulfate. C. P. General Chemical Company.

7. Anhydrone. Magnesium perchlorate. C. P. General Chemical Company.

8. Asbestos. Paper. Wilkens-Anderson Company.

9. Ascarite. General Chemical Company.

10. Benzoic acid. C. P. General Chemical Company.

11. p-Bromobenzoic acid. C. P. Eastman Kodak Company.

12. Congo Red. Hartman-Leddon Company.

One gram of Congo Red was dissolved in 100 ml. of distilled water.

13. Copper gauze. Forty mesh. Wilkens-Anderson Company.

14. Copper wire. Number sixteen. Wilkens-Anderson Company.

15. Cupric oxide. C. P. General Chemical Company.

16. Cupric oxide. Wire. C. P. General Chemical Company.

17. Glycerol. C. P. Wilkens-Anderson Company.

18. Gold chloride. C. P. American Platinum Works.

A 0.5 per cent solution was prepared by dissolving 0.25 g. of gold chloride in 100 ml. of distilled water.

19. Hydrazine hydrate. Eastman Kodak Company.

20. Hydrochloric acid. C. P. Sp. gr. 1.18 to 1.19. General Chemical Company.

A dilute solution was prepared and compared with 0.2078N sodium hydroxide using methyl orange as the indicator. Three determinations gave a mean of 0.2768 ± 0.0002N hydrochloric acid.

21. Hydrogen peroxide. Thirty per cent. Merck and Company.

A 1 per cent solution was prepared by dissolving 3.5 ml. of a 30 per cent solution of hydrogen peroxide in 100 ml. of distilled water.

22. Iodine. Resublimed. Merck and Company.

23. Lead acetate. C. P. General Chemical Company.

Five grams of lead acetate were dissolved in 100 ml. of distilled water.

24. Lead peroxide. C. P. General Chemical Company.

Four grams of lead peroxide were mixed thoroughly with 4 g. of Pb_3O_4, which was prepared by heating lead peroxide in a porcelain crucible at 400 to 450°C. (103).

25. Mercury. C. P. J. T. Baker and Company.

26. Methyl orange. Hartman-Leddon Company.

A 0.1 per cent solution was prepared by dissolving 0.1 g. of methyl orange in 100 ml. of distilled water.

27. α-Naphthol. C. P. General Chemical Company.

A 10 per cent solution of sodium hydroxide was added dropwise to a solution of 0.5 g. of α-naphthol in 100 ml. of distilled water until the α-naphthol was dissolved.

28. Nylon. A scoured fabric, filling rib weave, contained no sulfur, Huguet Silk Corporation, New York, 1941.

29. Oxygen. Pure. Balback Company.

30. Palladious chloride. C. P. American Platinum Works.

A 0.5 per cent solution was prepared by dissolving

0.25 g. of palladious chloride in 100 ml. of distilled water.

31. Paraffin. Standard Oil Company.

32. Petrolatum. Standard Oil Company.

33. Potassium persulfate. C. P. General Chemical Company.

Potassium pyrosulfate was prepared by heating potassium persulfate in a porcelain crucible to a temperature below a red heat until sulfur trioxide was evolved and frothing ceased (236b).

34. Potassium sulfide. C. P. J. T. Baker and Company.

Ten grams of potassium sulfide were dissolved in 100 ml. of water.

35. Potassium titanium oxalate. $K_2TiO(C_2O_4)_2 \cdot 2H_2O$. C. P. Eimer and Amend Company.

A standard solution of titanium sulfate was prepared by transferring 2.7582 g. of potassium titanium oxalate to a 500-milliliter Kjeldahl flask, adding 4 g. of ammonium sulfate and 50 ml. of concentrated sulfuric acid, and heating the mixture twenty minutes (208). The liquid was cooled, poured into 800 ml. of distilled water, again cooled, and diluted to 1 l. in a volumetric flask and filtered. One milliliter of this solution was tested for the complete destruction of oxalate by the addition of a drop of 1 per cent potassium permanganate.

This solution was standardized according to the procedure recommended by Thornton (207). The titanium in a measured volume of the solution of titanium sulfate was precipitated by <u>cupferron</u>; this precipitate was ignited and weighed as titanium dioxide. Four parallel determinations gave a mean of 0.000662 ±

0.000001 g. of titanium dioxide per milliliter.

36. Sodium. Metal. General Chemical Company.

37. Sodium hydroxide. C. P. General Chemical Company.

a. Forty grams of C. P. reagent were dissolved in 100 ml. of distilled water.

b. A dilute solution was prepared and compared with 0.2060N hydrochloric acid using methyl orange as indicator. Three determinations gave a mean of 0.2078 ± 0.0001N sodium hydroxide.

38. Sodium nitrite. C. P. General Chemical Company.

39. Sodium sulfate. Anhydrous. C. P. General Chemical Company.

40. Sulfuric acid. C. P. Sp. gr. 1.84. General Chemical Company.

41. Zinc. Dust. General Chemical Company.

42. Zinc. Mossy. General Chemical Company.

Analyses

Ash

A one-gram to three-gram sample was brought to constant weight, in a weighing bottle previously dried until constant, at 105 to 110°C. in an electric oven. It was then ignited to constant weight in a porcelain crucible, previously ignited until constant, at the dull red heat of an electric muffle furnace. Each weighing was made with a tare.

TABLE I. DETERMINATION OF TOTAL ASH

Determination	Fabric	Ash	
number	gram	gram	percentage of fabric
1A*	3.1171	0.0108	0.35
2A	3.0053	0.0101	0.34
3A	2.8055	0.0099	0.35
4A	2.9112	0.0102	0.35
5A	2.8321	0.0099	0.35
1B	1.6834	0.0061	0.36
2B	1.7039	0.0059	0.35
3B	1.7943	0.0061	0.34
4B	1.7660	0.0062	0.35
5B	1.7275	0.0063	0.37
Mean			0.35
Deviation			0.01

* Parallel determinations are lettered alike.

Attempted acetylation of nylon

Approximately 1.5 g. of nylon were refluxed seven hours with 75 ml. of glacial acetic acid and 0.2 g. of zinc dust (101). Then the solution was poured into 500 ml. of distilled water; the precipitate was stirred, washed, and filtered and the melting point of the dried product was determined to be 245°C.

Attempted diazotization of nylon

Approximately 2.5 g. of nylon were immersed in a solution of 200 ml. of water, 4 ml. of concentrated hydrochloric acid, and 10 g. of sodium nitrite (101). The nylon was worked in this bath at 5°C. for fifteen minutes, removed, rinsed in water at 5°C. and then worked in a bath of 2 per cent α-naphthol at 5°C.

The sample was removed, rinsed and dried at room temperature.

In three such attempts no diazotization of the fiber was observed.

Breaking strength and elongation of fabric at breaking load

Twenty warp and twenty filling specimens of the fabric were prepared for testing by the raveled-strip method and were conditioned for one week at 70 ± 2°F. and 65 ± 2 per cent R. H. before testing (172). A like number of each was immersed in distilled water at room temperature for one hour and then withdrawn singly and tested wet.

A Scott Universal Tester with autographic recorder was used to determine the breaking strength and elongation at breaking load of conditioned and wet warp and filling specimens. The two-inch jaws of the machine were clamped 3 in. apart on the one-inch strip, which was stretched under an initial load of six ounces. This initial load was removed before the jaws were started moving downward at a speed of 12 ± 0.5 in. per minute until the one-inch strip was strained to its breaking point (2).

Actual elongation at breaking load was measured to one-ninth inch on the autographically recorded stress-strain curve.

TABLE II. DETERMINATION OF BREAKING STRENGTH AND ELONGATION

Determination	Conditioned fabric					
	Breaking strength		Elongation		Breaking str	
	Warp	Filling	Warp	Filling	Warp	Filli
number	pound per inch	pound per inch	percentage	percentage	pound per inch	pound inch
1	96	41	30	30	84	32
2	95	42	33	32	83	34
3	101	42	37	30	85	34
4	99	41	37	30	82	34
5	102	41	37	30	80	34
6	96	41	33	30	83	34
7	98	41	--	30	81	34
8	98	41	--	32	82	34
9	97	41	--	30	72	36
10	97	41	--	30	82	33
11	96	41	--	32	78	34
12	96	41	--	30	79	34
13	83	41	30	30	81	34
14	96	41	30	30	83	36
15	99	41	32	30	72	34
16	84	41	30	32	74	34
17	95	41	28	30	78	34
18	99	41	30	30	74	33
19	96	41	33	30	78	34
20	97		30		70	33
21	95		32			
22	95		30			
23	83		26			
24	92		30			
25	89		30			
26	100		33			
Mean	95	41	32	30	79	34
Deviation	4	0	2	0	4	(

ETERMINATION OF BREAKING STRENGTH AND ELONGATION OF FABRIC

Conditioned fabric			Wet fabric			
strength	Elongation		Breaking strength		Elongation	
lling	Warp	Filling	Warp	Filling	Warp	Filling
und per ch	percentage	percentage	pound per inch	pound per inch	percentage	percentage
41	30	30	84	32	30	30
42	33	32	83	34	30	30
42	37	30	85	34	30	30
41	37	30	82	34	30	30
41	37	30	80	34	30	30
41	33	30	83	34	30	30
41	--	30	81	34	30	32
41	--	32	82	34	30	33
41	--	30	72	36	26	30
41	--	30	82	33	30	30
41	--	32	78	34	28	32
41	--	30	79	34	28	30
41	30	30	81	34	30	30
41	30	30	83	36	30	32
41	32	30	72	34	30	30
41	30	32	74	34	28	33
41	28	30	78	34	28	30
41	30	30	74	33	28	30
41	33	30	78	34	30	30
	30		70	33		30
	32					
	30					
	26					
	30					
	30					
	33					
41	32	30	79	34	29	31
0	2	0	4	0	1	1

Dichroism

A sample of nylon was (1) dyed with Alizarin Blue S, (2) dyed with Congo Red, (3) mordanted with a solution of potassium dichromate, 10 per cent the weight of the sample and 0.125N as to hydrochloric acid, (4) mordanted in the same way and then dyed with Alizarin Blue S, (5) treated with auric chloride and suspended over hydrazine hydrate (88), or (6) treated with 0.1N iodine. Each treated sample was mounted in glycerol on a glass slide and examined under polarized light to determine whether or not dichroism was exhibited (177).

TABLE III. DICHROISM OF TREATED NYLON

Treatment			Dichroism
Reagent	Temperature	Time minute	
1.0 per cent Congo Red	boiling	30	positive
0.5 per cent auric chloride followed by hydrazine hydrate	room	15	positive
0.1N iodine	40°C.	540	slightly dichroic
1.0 per cent Alizarin Blue S	boiling	30	negative
10 per cent potassium dichromate on the weight of the fabric and 0.125N as to hydrochloric acid	boiling	60	negative
10 per cent potassium dichromate, 0.125N as to hydrochloric acid, for 60 min. boiling and then 1.0 per cent Alizarin Blue S	boiling	60	negative

Distribution of yarns by number

The warp and filling yarns were counted at ten different places in the fabric (2).

TABLE IV. DISTRIBUTION OF YARNS BY NUMBER

Determination number	Yarn count: Warp yarn number per inch	Yarn count: Filling yarn number per inch
1	235	104
2	236	104
3	235	103
4	236	104
5	235	104
6	236	103
7	237	103
8	237	103
9	238	104
10	236	104
Mean	236	104
Deviation	1	0

Distribution of yarns by weight

Four two-inch squares were conditioned, weighed, and raveled into warp and filling yarns, and each kind of yarn was weighed separately on a watch glass.

TABLE V. DISTRIBUTION OF YARNS BY WEIGHT

Determination number	Fabric	Warp yarn		Filling yarn	
	gram	gram	percentage of fabric	gram	percentage of fabric
1	0.1752	0.1227	70.03	0.0527	30.08
2	0.1772	0.1240	69.98	0.0536	30.25
3	0.1758	0.1231	70.02	0.0529	30.09
4	0.1757	0.1232	70.12	0.0531	30.21
Mean			70.04		30.16
Deviation			0.04		0.07

Melting point

The melting point of the fabric was determined both macroscopically and microscopically. Concentrated sulfuric acid containing anhydrous sodium sulfate was placed in a Thiele tube and the thermometer with a melting-point tube attached was lowered into this medium. Heat was applied at the side-arm of the Thiele tube and the melting point of the substance in the capillary tube was observed. The thermometer was calibrated by determining the melting point of p-bromobenzoic acid: observed, 248-9°C.; literature, 251°C. (194). Three determinations of the melting point of nylon macroscopically gave a mean of 248.8 ± 0.6°C. Two determinations microscopically gave a mean of 250.5° ± 0.5°C. In all cases the fiber turned from white to light tan at 229°C.

Microscopic examination

A polarizing microscope in which light was traveling parallel through the system was used in studying the fiber. Its double refraction was high; it appeared green inside and red at the periphery when viewed between crossed Nicol prisms. The fiber exhibited parallel extinction, i.e. extinction when the lengthwise direction of the fiber was lying in the direction of the polarizer.

The refractive indices were proved different by the Becke-line method, for which the fiber was immersed in liquids each of known refractive index (238). The mounted fiber was rotated with the stage until light was not doubly refracted but came through the crystal in the preferred direction. The refractive index observed for the ray vibrating lengthwise in the fiber was 1.501 to 1.550; since the larger index of refraction is parallel to the longer axis of the crystal, nylon is described as a positive fiber. The fiber exhibited retardation equal to the second order on the quartz wedge between blue and green.

Moisture

Four five-gram samples of the fabric were conditioned and weighed after a period of one week at 70 ± 2°F. and 65 ± 2 per cent R. H. Each was then dried to constant weight, in a weighing bottle previously dried until constant, at 105-110°C. in an electric oven. The percentage of moisture was computed on the weight of the conditioned sample.

TABLE VI. DETERMINATION OF MOISTURE

Determination number	Weight of sample: Conditioned gram	Weight of sample: Dried, 105-110°C. gram	Moisture percentage of conditioned fabric
1	4.8508	4.7042	3.02
2	4.8530	4.7092	2.96
3	4.8256	4.6831	2.95
4	4.8519	4.7054	3.02
5	4.8850	4.7422	2.92
Mean			2.97
Deviation			0.04

Qualitative tests for sulfur

Yarns from different parts of the fabric were cut into small pieces and mixed thoroughly before a portion was dropped on molten sodium in a small test tube which was heated strongly before the tube was plunged into 20 ml. of distilled water. The solution was boiled, filtered, and made acidic with acetic acid before the dropwise addition of a solution of lead acetate. Since no precipitate of lead sulfide formed, this test was negative.

On a micro scale (238) small pieces of yarns were heated with zinc dust in a tiny pyrex tube closed at one end. The cooled zinc dust was emptied on a glass slide, a ring of glass tubing was placed around it, and dilute hydrochloric acid was added. Immediately another half-slide on which was a drop of ammoniacal lead acetate was inverted over the reacting mixture for a few minutes. Then the solution on the upper slide was examined microscopically for the formation of a precipitate of lead sulfide. In three such determinations no positive test for sulfur was obtained.

Quantitative estimation of carbon and hydrogen

The apparatus and procedure for the determination of carbon and hydrogen have been described by Fisher (103). Since a pressure gauge for the oxygen tank was not available, it was necessary to store oxygen in a 20-liter bottle and adjust the flow of oxygen by means of a pressure regulator as recommended by Gattermann and Wieland (112). The combustion tube was fitted with a tube containing 8 g. of a 1:1 mixture of PbO_2 and Pb_3O_4 which was kept at 300 to 320°C. by means of a third section of the furnace in order to absorb the oxides of nitrogen formed during the combustion (103).

The combustion train consisted of a storage bottle for oxygen, a pressure regulator, a bubble counter, a pre-combustion tube, an absorption bottle containing Anhydrone, Ascarite, and then Anhydrone, the filled combustion tube (59), an absorption tube filled with Anhydrone, another filled with Ascarite, a guard tube filled with Anhydrone, and finally a bottle of 0.5 per cent palladious chloride.

The tube was burned out for two hours without connecting the absorption tubes to the train. Then some benzoic acid was burned in the apparatus with the absorption tubes connected to the train. The tubes were brought to constant weight in the manner described by Fisher (103).

Acetanilide and nylon, the ravelings from two yards of the fabric, were each mixed with anhydrous cupric oxide and burned separately in the apparatus. A low flame was used for the first heating and the small section of the three-sectioned furnace for the final heating. Between each two determinations the absorption tubes were connected to the train for forty minutes and weighed to ascertain if they were still constant.

TABLE VII. DETERMINATION OF CARBON AND HYDROGEN

Substance	Weight of sample	Water	Hydrogen	Carbon dioxide	Carbon
gram	gram	gram	percentage of compound*	gram	percentage of compound*
Acetanilide	0.1963	0.1202	6.85[a]	0.5116	71.12[b]
Fabric					
1	0.1793	0.1561	9.77	0.4165	63.61
2	0.1737	0.1494	9.66	0.4010	63.22[x]
3	0.1887	0.1702	10.13[x]	0.4384	63.64
4	0.1664	0.1449	9.78	0.3859	63.52
5	0.2147	0.1871	9.79	0.4975	63.47
Mean			9.75[c]		63.56[d]
Deviation			0.05		0.07

* The weight of the fabric was corrected for 0.35 per cent of ash.

[a] Theoretical value, 6.71

[b] Theoretical value, 71.09

[c] Theoretical value, 9.80

[d] Theoretical value, 63.68

[x] Rejected observation

Quantitative estimation of nitrogen

A sample of nylon constant in weight at 105-110°C. was converted into a colorless solution by 25 ml. of boiling concentrated sulfuric acid containing a drop of mercury and 10 g. of anhydrous sodium sulfate. After cooling and diluting the solution with 200 ml. of distilled water, a small piece of paraffin, 100 ml. of 40 per cent sodium hydroxide, 20 ml. of 10 per cent potassium sulfide, and a few small pieces of mossy zinc were added and the mixture was distilled immediately into 75.00 ml. of standard hydrochloric acid. The excess of acid was titrated with a standard solution of sodium hydroxide using methyl orange as indicator.

TABLE VIII. DETERMINATION OF TOTAL NITROGEN

Determination number	Fabric of nylon gram	Sodium hydroxide 0.2078N milliliter	Nitrogen percentage of nylon*
1	none	99.95	0.00
2	1.4202	41.33	12.06
3	1.3987	42.24	12.05
4	1.4215	41.97	11.91[x]
5	1.4389	40.63	12.04
6	1.6680	30.84	12.10
7	1.6523	31.37	12.12
8	1.6649	31.04	12.09
Mean			12.08**
Deviation			0.03

[x] Rejected observation
* Corrected for 0.35 per cent of ash in the fabric
** Theoretical value, 12.38

Quantitative estimation of titanium

The ash obtained from nylon was fused in a porcelain crucible with approximately 2 g. of potassium pyrosulfate. The melt was cooled, 10 ml. of concentrated sulfuric acid were added, and the mass was heated until it liquefied. This liquid was dissolved in 25 ml. of cold water and transferred to a 100-milliliter or 250-milliliter volumetric flask. In the case of solutions to be diluted to 250 ml., 8 ml. more of sulfuric acid were added. Then 10 ml. of 1 per cent hydrogen peroxide were added and the solution was diluted to volume.

The amount of titanium dioxide per milliliter was determined colorimetrically according to the Nessler procedure (236a) and also by means of a two-celled photoelectric colorimeter. The standard solutions for comparison were made by measuring a definite volume of the standard titanium sulfate in a microburette, adding the required quantity of concentrated sulfuric acid and hydrogen peroxide, and making the solution up to volume either in a Nessler tube or in a 100-milliliter volumetric flask.

A two-celled photoelectric colorimeter, the Wilkens-Anderson KWSZ Photometer loaned by the Biophysical Laboratory, was standardized by determining the percentage of light transmitted by a series of peroxidized standard solutions, using distilled water as the reference liquid (235).

TABLE IX. DETERMINATION OF TITANIUM BY THE NESSLER METHOD

Determination	Fabric	Ash		Equivalent of standard solution (0.000662 g. of TiO_2 per milliliter)	Titanium dioxide		
number	gram	gram	percentage of nylon	milliliter	gram	percentage of fabric	percentage of ash
1	3.1171	0.0108	0.35	13.20	0.0087	0.28	80.6[x]
2	3.0053	0.0101	0.34	13.20	0.0087	0.29	86.1
3	2.8055	0.0099	0.35	12.91	0.0085	0.30	85.9
4	2.9112	0.0102	0.35	13.20	0.0087	0.30	85.3
5	2.8321	0.0099	0.35	12.91	0.0085	0.30	85.9
Mean			0.35			0.29	85.8
Deviation			0.00			0.01	0.2

[x] Rejected observation

TABLE X. THE STANDARDIZATION OF THE KWSZ PHOTOMETER

Volume of standard titanium sulfate diluted to 100 ml. milliliter	Titanium dioxide per 100 ml. gram	Transmission percentage
1	0.000662	74.5
3	0.001986	49.9
5	0.003310	33.9
7	0.004534	22.6
9	0.005958	14.9
10	0.006620	12.3
11	0.007282	10.1
13	0.008606	6.2
15	0.009930	3.7

Two series of filters, Corning 511 Violet (5.0 mm. thick) with Corning 306 Noviol O Shade (2.0 mm. thick), were found to yield better results than the Corning 511 with Noviol A Shade 038 filter which was recommended by Dahle for the determination of fluorine by the peroxidized titanium method (93). Using a peroxidized titanium solution containing 15 ml. of standard titanium sulfate per 100 ml., filter 511 and 038 gave a reading in transmission of 20 per cent, while filter 511 and 306 gave a reading of 17 per cent. The latter filter was chosen for use in subsequent determinations of titanium; the maximum absorption of the solution of peroxidized titanium is near 4100Å, and this filter isolates that region more readily than the filter recommended by Dahle.

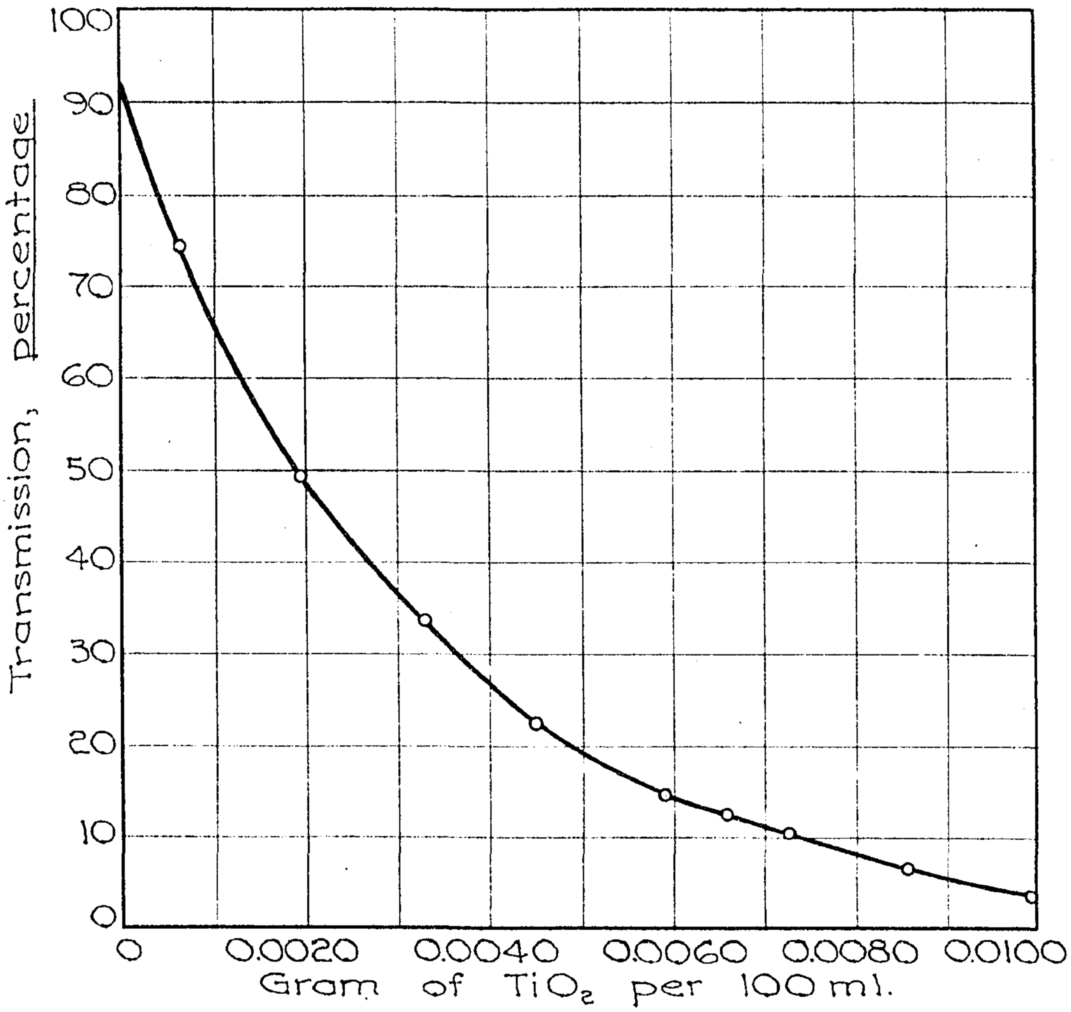

FIG. 1. GRAPH FOR CALIBRATION OF THE KWSZ PHOTOMETER.

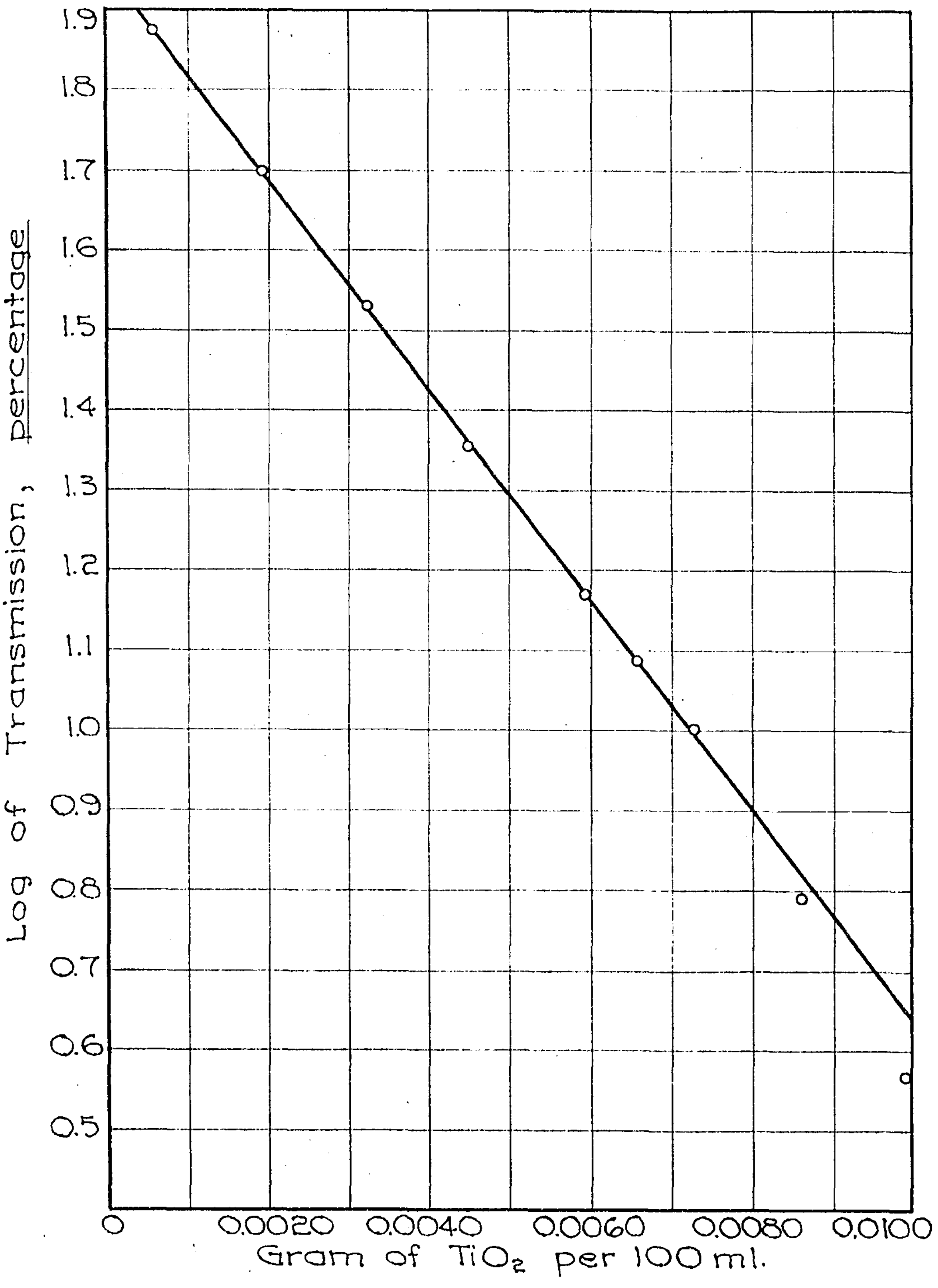

FIG. 2. THE RELATIONSHIP BETWEEN THE WEIGHT OF TiO_2 PER 100 ML. AND THE LOG OF THE TRANSMISSION.

In the manipulation of the KWSZ Photometer the same procedure was rigorously followed each time. The instrument was allowed to come to equilibrium for fifteen minutes with water running through the cooling system around the photocell; both sets of filters were in place and one of the special test tubes containing distilled water was in the path of the light. Then the instrument was set on 100 per cent transmission for distilled water. The solution of peroxidized titanium was placed in another special test tube and shifted into place. The percental transmission was read after so adjusting the controls that the galvanometer registered zero.

TABLE XI. DETERMINATION OF TITANIUM WITH THE KWSZ PHOTOMETER.

Determination	Fabric	Ash		Transmission	Titanium dioxide			
					Interpolation	per 100 ml.		
number	gram	gram	percentage of fabric	percentage	gram	gram	percentage of fabric	percentage of ash
1	1.6834	0.0061	0.36	21.1	0.004811	0.0048	0.29	79
2	1.7039	0.0059	0.35	50.1*	0.001975	0.0049	0.29	83
3	1.7943	0.0061	0.34	50.4*	0.001959	0.0049	0.27	80
4	1.7660	0.0062	0.35	19.8	0.005052	0.0051	0.29	82
5	1.7275	0.0063	0.37	50.4*	0.001959	0.0049	0.28	78
Mean			0.35				0.28	80
Deviation			0.01				0.01	2

* These solutions were diluted to 250 ml.

Thickness of fabric

Ten measurements of thickness at different portions of the fabric were made by means of an automatic micrometer which pressed upon a circle of the fabric 3/16 in. in diameter with a pressure of 6 ounces.

TABLE XII. THICKNESS OF FABRIC

Determination number	Thickness inch
1	0.0036
2	0.0036
3	0.0035
4	0.0035
5	0.0034
6	0.0034
7	0.0035
8	0.0034
9	0.0036
10	0.0036
Mean	0.0035
Deviation	0.0001

Twist of yarn

The twist of warp and filling yarns was determined with an Improved United States Testing Company Tester according to the procedure recommended by the American Society for Testing Materials (2, 3). Each type of yarn conformed to a Z twist, since the direction of the spirals was the same as the slant of the middle line in the letter Z. The twist of filling yarns was determined with 10-inch lengths, that of warp yarns with 4-inch lengths.

TABLE XIII. TWIST OF YARN

Determination number	Warp yarn number per four inches	Filling yarn number per ten inches
1	70	168
2	75	119
3	71	134
4	65	164
5	57	127
6	68	123
7	75	167
8	72	180
9	61	132
10	78	190
11	71	172
12	72	165
13	91	170
14	65	157
15	78	154
16	70	161
17	58	137
18	78	119
19	57	114
20	74	140
21	59	160
22	68	
Mean	69	150
Deviation	6	20
Mean number per inch	17	15
Deviation	2	2

Weight of fabric

Four samples were cut 4 in. in length and the entire width of the fabric, measured accurately, conditioned, and weighed (2).

TABLE XIV. WEIGHT OF FABRIC

Determination number	Fabric gram	Length inch	Width inch	Weight ounce per square yard
1	7.0294	4.03	39.94	2.00
2	7.0447	4.03	39.94	2.00
3	7.1039	4.06	39.91	2.00
4	7.0256	4.03	39.91	2.00
5	7.0571	4.01	39.91	2.01
Mean				2.00
Deviation				0.00

Yarn number

Four 10-yard lengths of each kind of yarn were conditioned and weighed and each yarn number was computed as thousands of yards per pound (2).

TABLE XV. YARN NUMBER

Determination	Warp yarn		Filling yarn	
number	gram	typp	gram	typp
1	0.0472	96.1	0.0466	97.3
2	0.0472	96.1	0.0463	98.0
3	0.0475	95.5	0.0467	97.1
4	0.0475	95.5	0.0467	97.1
Mean		95.8		97.4
Deviation		0.3		0.3

EXPERIMENTAL PROCEDURE

Materials

1. Alkali alkyl sulfate (87).

A 0.25 per cent solution was prepared by dissolving 0.25 g. of an alkali alkyl sulfate in 100 ml. of distilled water.

2. Aluminum sulfate. $Al_2(SO_4)_3 \cdot 18H_2O$. Analytical reagent. Mallinckrodt Chemical Works.

3. Arsenic oxide. 99.95 per cent. General Chemical Company.

A solution 0.1000N as to arsenic oxide was prepared by dissolving 4.9480 g. of arsenic oxide in 40 ml. of 1N sodium hydroxide; this solution was made acid to litmus with 1N sulfuric acid and was diluted to 1 l. in a volumetric flask.

4. Bordeaux. Colour Index No. 88. Hartman-Leddon Company.

A 0.2 per cent solution of this indicator was prepared by dissolving 0.2 g. of Bordeaux in 100 ml. of distilled water.

5. Carbon electrodes. National Projector No. 013 and No. 148. Coluvbia National Carbon Company.

6. Formaldehyde. 37.0 per cent by weight. General Chemical Company.

A 1 per cent solution of formaldehyde was prepared and standardized by oxidizing 25 ml. of the solution with hydrogen peroxide in the presence of 100 ml. of 0.2139N sodium hydroxide and titrating the excess sodium hydroxide with 0.2268N hydrochloric acid using litmus as indicator (57, 192).

7. HTH. Active ingredient $Ca(OCl)_2$, 70 per cent. The Mathieson Alkali Works.

An approximately 0.3<u>N</u> solution of calcium hypochlorite was prepared by dissolving the calculated quantity of HTH in distilled water. Baths of 50 ml. of solution per gram of fabric were prepared; these baths and Erlenmeyer flasks containing approximately 150 ml. each of calcium hypochlorite were placed in a constant-temperature bath for one hour. Then samples were put into their respective solutions and one blank was removed and standardized by pipeting 25 ml. of the standard arsenious acid into an Erlenmeyer flask, adding 1 g. of potassium bromide and 0.5 g. of sodium hydrogen carbonate and titrating this solution with the calcium hypochlorite until within a few milliliters of the end point, then adding one drop of <u>Bordeaux</u> indicator and titrating dropwise until the pink color of the indicator changed to a very faint yellow or green (138). Whenever a sample was removed from the constant-temperature bath, a blank was removed and titrated according to this procedure but no change in concentration was observed.

Only one solution of calcium hypochlorite was prepared. Three standardizations of three determinations each over a period of three months gave means of 0.2941 ± 0.0001<u>N</u>, 0.2906 ± 0.0002<u>N</u>, and 0.2875 ± 0.0001<u>N</u> calcium hypochlorite.

8. Hydrogen peroxide. Thirty per cent. Merck and Company.

A 3 per cent solution was prepared by dissolving 10.5 ml. of 30 per cent hydrogen peroxide in 100 ml. of distilled water.

9. Hydrochloric acid. C. P. Sp. gr. 1.18 to 1.19. General Chemical Company.

A dilute solution was prepared and standardized against sodium carbonate using methyl orange as indicator. Six determinations gave a mean of 0.2268 ± 0.0002N hydrochloric acid.

10. Iodine. Resublimed. Merck and Company.

A dilute solution of iodine was prepared by dissolving 25 g. of potassium iodide in a little water, adding 12.7 g. of iodine, shaking, and diluting to 1 l. The solution was standardized against standard arsenite using starch as the indicator (117c). Three determinations gave a mean of 0.1029 ± 0.0000N iodine.

11. Iron wire. Purity 99.8 per cent. Merck and Company.

12. Mercuric chloride. C. P. General Chemical Company.

Twenty-seven grams of mercuric chloride were dissolved in 1 l. of distilled water.

13. Potassium bromide. C. P. General Chemical Company.

14. Potassium chloride. C. P. General Chemical Company.

A saturated solution was prepared for use as a bridge in the determination of pH.

15. Potassium dichromate. C. P. General Chemical Company.

a. Two 0.1N solutions were prepared by dissolving 4.903 g. of potassium dichromate in water and diluting the solution to 1 l. in a volumeteric flask. These solutions were standardized against iron wire of known purity using potassium ferricyanide as outside indicator (117a). Four determinations in each case gave a mean of 0.1009 ± 0.0001N and 0.1004 ± 0.0001N for the two solutions.

b. A solution approximately 0.04N was prepared and standardized in the same manner. Three determinations gave a mean of 0.04231 ± 0.0001N potassium dichromate.

16. Potassium ferricyanide. C. P. General Chemical Company.

17. Phenol. Carbolic acid. Merck and Company.

One gram was dissolved in 100 ml. of distilled water.

18. Potassium hydrogen phthalate. C. P. General Chemical Company.

A 0.05M solution, pH 4.0, was prepared by adding 0.40 ml. of 0.1N sodium hydroxide to 50 ml. of 0.1M potassium hydrogen phthalate and diluting this solution to 100 ml.

19. Potassium iodide. C. P. General Chemical Company.

20. Potassium permanganate. C. P. General Chemical Company.

An approximately 0.2N solution was prepared by dissolving 6.4 g. of potassium permanganate per liter of water, boiling the solution fifteen minutes, allowing the solution to stand overnight and filtering it through asbestos. This solution was standardized against sodium oxalate (117b). Six determinations gave a mean of 0.2040 ± 0.0001N potassium permanganate.

21. Scotch tape. Local distributor.

22. Sodium hydrogen carbonate. Powder. Reagent. General Chemical Company.

23. Sodium hydrogen sulfite. Anhydrous powder. Reagent. General Chemical Company.

Approximately 0.15N and 0.3N solutions of sodium hydrogen sulfite were prepared and standardized against a solution of standard potassium permanganate containing 3 g. of potassium iodide and 5 ml. of concentrated hydrochloric acid. After this solution had stood in the dark three minutes, it was titrated with a solution of sodium hydrogen sulfite, using starch as the indicator (210). Four determinations in each case gave a mean of 0.1757 ± 0.0001N and 0.3003 ± 0.0002N for the two solutions.

24. Sodium carbonate. C. P. General Chemical Company.

25. Sodium hydroxide. C. P. General Chemical Company.

Two solutions were prepared and standardized against 0.2268N hydrochloric acid using methyl orange as indicator. Four determinations gave a mean of 0.2139 ± 0.0001N for one solution and three determinations gave a mean of 0.2975 ± 0.0001N for the other.

26. Sodium oxalate. C. P. Powder for standardizations. General Chemical Company.

27. Stannous chloride. C. P. General Chemical Company.

Fifty grams of stannous chloride were dissolved in 100 ml. of concentrated hydrochloric acid.

28. Starch. Potato. General Chemical Company.

A thin paste of starch and cold water was made and this was rinsed into 25 ml. of boiling water.

29. Sulfuric acid. C. P. Sp. gr. 1.84. General Chemical Company.

Two solutions of sulfuric acid were prepared and standardized against 0.2139N sodium hydroxide using methyl orange as indicator. In each case three comparisons gave a mean of 0.1888 ± 0.0001N for one solution and 1.0279 ± 0.0003N for the other.

Mordanting

Preparation of samples

Before weighing the one-gram to two-gram samples of fabric it was necessary to ravel yarns back from the edge at least a quarter of an inch and to whip the edges securely with a nylon yarn from the fabric so that the samples would remain intact during treatment.

The breaking-strength specimens were cut 1.3 in. wide and 7 in. in the filling direction and three filling yarns were drawn on either side of the central inch. After the samples were treated, the yarns left at either side of the central inch were removed before the specimen was broken wet.

Determination of pH

Baths of each mordanting concentration were prepared, shaken vigorously, and allowed to stand at room temperature for one hour.

The pH of each solution was determined at room temperature by means of a Cameron pH-Meter in the Biophysical Laboratory. The glass electrode was calibrated against 0.05M potassium hydrogen phthalate, pH 4.0. The pH of each solution after mordanting was also determined at room temperature.

Treatment of the fabric with aluminum sulfate

A weighed sample of the fabric was placed in a 250-milliliter Erlenmeyer flask containing fifty volumes of distilled water and an amount of aluminum sulfate such that there were 0.40, 0.50, 1.50, 3.00 or 6.00 g. of aluminum oxide present per gram of fabric. The Erlenmeyer flasks were put into a boiling water bath and were fitted with reflux condensers; the solutions were allowed to start refluxing and were refluxed for one hour. The samples were removed, rinsed for five three-minute periods in distilled water, and dried at room temperature before they were ashed. The initial and final pH of each bath was measured with a Cameron pH-Meter.

Treatment of the fabric with potassium dichromate

Fifty volumes of distilled water per gram of nylon, a bath of fifty volumes of 0.1N hydrochloric acid, a bath of fifty volumes of potassium dichromate, 4, 10, or 25 per cent the weight of the fabric, and a bath of potassium dichromate, 10 per cent the weight of the fabric and 0.025, 0.05, 0.075, 0.10, or 0.125N as to hydrochloric acid or 0.10N as to sodium hydroxide, were measured into 125-milliliter Erlenmeyer flasks. A sample was

TABLE XVI. EFFECT OF FIFTY-VOLUME BATHS OF ALUMINUM IN ONE HOUR AT 100°C. ON THE TOTAL ASH O

Determination	Mordanting Bath				Fabr
	Initial conditions		Equilibrial conditions		
	Aluminum sulfate			Exhaustion	
number	gram of Al_2O_3 per gram of fabric	pH	pH	percentage	gram
1*	0.40	3.2	3.2	0.10	1.8
1	0.50	3.0	3.1		1.0
2		3.0	3.1		1.0
Mean		3.0	3.1	0.26	
Deviation		0.0	0.0		
1	1.50	2.7	2.8		1.0
2		2.7	2.8		1.0
Mean		2.7	2.8	0.05	
Deviation		0.0	0.0		
1	3.00	2.5	2.4		1.1
2		2.5	2.4		1.1
3		2.5	2.6		1.0
4		2.5	2.4		1.1
5		2.5	2.4		1.0
Mean		2.5	2.4	0.01	
Deviation		0.0	0.1		
1		2.0	2.1		1.1
2	6.00	2.1	2.0		1.1
3		1.9	2.0		1.1
4		1.9	2.0		1.0
5		1.9	2.0		1.1
Mean		1.9	2.0	0.002	
Deviation		0.1	0.0		

---

* 100-volume bath.

BLE XVI. EFFECT OF FIFTY-VOLUME BATHS OF ALUMINUM SULFATE IN ONE HOUR AT 100°C. ON THE TOTAL ASH OF THE FABRIC.

Mordanting Bath				Fabric	Total ash	
ial conditions		Equilibrial conditions				
ulfate			Exhaustion			
2O3 per bric	pH	pH	percentage	gram	gram	percentage of fabric
	3.2	3.2	0.10	1.8371	0.0064	0.35
	3.0	3.1		1.0292	0.0046	0.45
	3.0	3.1		1.0108	0.0042	0.42
	3.0	3.1	0.26			0.44
	0.0	0.0				0.02
	2.7	2.8		1.0133	0.0039	0.38
	2.7	2.8		1.0740	0.0042	0.39
	2.7	2.8	0.05			0.38
	0.0	0.0				0.01
	2.5	2.4		1.1753	0.0037	0.31
	2.5	2.4		1.1321	0.0034	0.30
	2.5	2.6		1.0563	0.0033	0.31
	2.5	2.4		1.1055	0.0045	0.41
	2.5	2.4		1.0420	0.0041	0.39
	2.5	2.4	0.01			0.34
	0.0	0.1				0.04
	2.0	2.1		1.1081	0.0032	0.29
	2.1	2.0		1.1168	0.0033	0.30
	1.9	2.0		1.1465	0.0041	0.36
	1.9	2.0		1.0955	0.0035	0.32
	1.9	2.0		1.1015	0.0038	0.34
	1.9	2.0	0.002			0.32
	0.1	0.0				0.02

put into each flask; the flask was connected to a reflux condenser and was placed in a boiling water bath. The bath containing the fabric was brought to refluxing and refluxed for one hour. The nylon was removed, rinsed for five three-minute periods in 100 volumes of distilled water and dried at room temperature. Samples so treated were used in the determination of ash and, in the case of nylon treated with 10 per cent potassium dichromate which was 0.125N as to hydrochloric acid, in the determination of residual weight and nitrogen.

Ten filling specimens of the fabric were treated at the same concentrations in 250-milliliter Erlenmeyer flasks, rinsed for five three-minute periods in 100 volumes of distilled water, and broken wet after one hour in fifty volumes of distilled water. The volume of each mordanting bath was based on the oven-dry weight of each set of ten specimens. This value was determined by multiplying the air-dry weight of ten specimens by the factor (oven-dry weight/air-dry weight) derived from the alternate weighing in air of a set of ten breaking-strength samples and a one-gram to two-gram sample later dried to constant weight at 105 to 110°C. This procedure was followed in determining the factor for each of three sets of 100 breaking-strength specimens; twenty determinations in each case gave a mean of (1) 0.9814 ± 0.0007, (2) 0.9874 ± 0.0003, and (3) 0.9788 ± 0.0006. Another set of 100 specimens was conditioned and weighed, and each oven-dry weight was calculated by correcting for the percentage of moisture in the fabric at 70 ± 2°F. and 65 ± 2 per cent R. H.

TABLE XVII. EFFECT OF FIFTY-VOLUME BA
ONE HOUR AT 100°C. ON THE
NITROGEN AND WET STRENGTH

Determination	Mordanting bath				
	Initial condition			Equilibrial c	
	Potassium dichromate	Hydrochloric acid			Exhaustion
number	gram of Cr_2O_3 per gram of fabric	normality	pH	pH	percentage
1	none	none	5.9	6.4	
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
Mean					
Deviation					
1	0.0207	none	4.8	5.2	
2					
3					
4					
5					
6					
7					
8					
9					
Mean					4.34
Deviation					
Percental decrease					

IFTY-VOLUME BATHS OF POTASSIUM DICHROMATE IN
100°C. ON THE WEIGHT, CHROMIC OXIDE, TOTAL
D WET STRENGTH OF THE FABRIC.

		Fabric				
Equilibrial condition			Residual weight		Ash	
Exhaustion						
percentage	gram of Cr_2O_3 per gram of fabric	gram	gram	percentage of fabric	gram	perce of fa
		1.1594			0.0032	0.2
		1.1342			0.0031	0.2
		1.5235			0.0060	0.3
		1.1851			0.0046	0.3
		1.0494			0.0037	0.3
		1.0530			0.0037	0.3
		1.0233			0.0032	0.3
		1.0248			0.0032	0.3
		1.9218			0.0057	0.3
		1.9290			0.0057	0.3
		1.9394			0.0047	0.2
		1.8966			0.0047	0.2
		1.9940			0.0053	0.2
						0.3
						0.
		1.2213			0.0049	0.
		1.2696			0.0049	0.
		1.2370			0.0049	0.
		1.2744			0.0050	0.
4.34	0.0198					0.
	0.0000					0.

	Mordanted fabric					
t	Ash		Chromic oxide	Sodium hydroxide 0.2139N	Nitrogen	Breaking strength of wet filling
ge c	gram	percentage of fabric	gram per gram of fabric	milliliter	percentage of nylon	pounds per inch
	0.0032	0.28				33
	0.0031	0.27				31
	0.0060	0.39				32
	0.0046	0.39				32
	0.0037	0.35				33
	0.0037	0.35				31
	0.0032	0.31				31
	0.0032	0.31				32
	0.0057	0.30				30
	0.0057	0.30				32
	0.0047	0.24				
	0.0047	0.25				
	0.0053	0.27				
		0.31				32
		0.04				1
	0.0049	0.40				32
	0.0049	0.39				32
	0.0049	0.40				34
	0.0050	0.40				31
						30
						30
						34
						32
						36
		0.40	0.0009			32
		0.01	0.0000			1
						0

Determination	Mordanting bath				
	Initial condition			Equilibria	
	Potassium dichromate	Hydrochloric acid			Exhaustio
number	gram of Cr_2O_3 per gram of fabric	normality	pH	pH	percentag
1	0.0517	none	4.6	5.0	
2					
3					
4					
5					
6					
7					
8					
9					
10					
Mean					2.71
Deviation					
Percental decrease					
1		none	10.8*	11.0*	
2					
3					
4					
5					
6					
7					
8					
9					
Mean					3.29
Deviation					
Percental decrease					
1		0.001	3.2	4.0	
2					
3					
4					
5					
6					
7					
8					
9					
10					

---

* Sodium hydroxide was added so that the solution was 0.1<u>N</u>; the pH was de

TABLE XVII. (continued)

Equilibrial condition		Fabric	Mor			
Exhaustion			Residual weight		Ash	
percentage	gram of Cr_2O_3 per gram of fabric	gram	gram	percentage of fabric	gram	percent of fabr
		1.8469			0.0083	0.45
		1.7456			0.0076	0.44
		1.6764			0.0073	0.44
		1.6750			0.0078	0.47
2.71	0.0503 0.0001					0.45 0.01
		2.0148			0.0100	0.50
		2.0114			0.0096	0.48
		1.9940			0.0094	0.47
		1.9933			0.0093	0.47
3.29	0.0500 0.0001					0.48 0.01
		1.0680			0.0064	0.60
		1.0418			0.0067	0.64
		1.0695			0.0065	0.61
		1.9041			0.0105	0.55

e pH was determined with pH paper.

Mordanted fabric						
weight	Ash		Chromic oxide	Sodium hydroxide 0.2139N	Nitrogen	Breaking strength of wet filling
centage fabric	gram	percentage of fabric	gram per gram of fabric	milliliter	percentage of nylon	pounds per inch
	0.0083	0.45				31
	0.0076	0.44				30
	0.0073	0.44				30
	0.0078	0.47				34
						33
						32
						32
						30
						30
						28
		0.45	0.0014			31
		0.01	0.0001			1
						3
	0.0100	0.50				28
	0.0096	0.48				31
	0.0094	0.47				33
	0.0093	0.47				33
						32
						31
						31
						30
						33
		0.48	0.0017			31
		0.01	0.0001			1
						3
	0.0064	0.60				32
	0.0067	0.64				33
	0.0065	0.61				31
	0.0105	0.55				32
						32
						31
						32
						32
						31
						32

Determination	Mordanting bath				
	Initial condition			Equilibrian	
	Potassium dichromate	Hydrochloric acid			Exhaustion
number	gram of Cr_2O_3 per gram of fabric	normality	pH	pH	percentage
Mean					5.61
Deviation					
Percental decrease					
1		0.025	1.5	1.7	
2					
3					
4					
5					
6					
7					
8					
9					
Mean					18.8
Deviation					
Percental decrease					
1		0.05	1.2	1.3	
2					
3					
4					
5					
6					
7					
8					
9					
Mean					30.9
Deviation					
Percental decrease					
1		0.075	1.0	1.0	
2					
3					
4					
5					
6					
7					

TABLE XVII. (continued)

g bath			Fabric				
Equilibrian condition				Residual weight		As	
	Exhaustion						
pH	percentage	gram of Cr_2O_3 per gram of fabric	gram	gram	percentage of fabric	gram	pe of
	5.61	0.0488 0.0003					
1.7			1.3140			0.0167	
			1.0016			0.0133	
			1.0043			0.0131	
			1.1610			0.0149	
			1.1973			0.0149	
	18.8	0.0420 0.0002					
1.3			1.1981			0.0228	
			1.0803			0.0201	
			1.1280			0.0212	
			1.2289			0.0242	
			1.2522			0.0245	
	30.9	0.0357 0.0004					
1.0			1.0978			0.0267	
			1.0856			0.0269	
			1.0355			0.0255	
			1.0994			0.0272	

Mordanted fabric						
al weight	Ash		Chromic oxide	Sodium hydroxide 0.2139N	Nitrogen	Breaking strength of wet filling
percentage of fabric	gram	percentage of fabric	gram per gram of fabric	milliliter	percentage of nylon	pounds per inch
		0.60	0.0029			32
		0.03	0.0003			0
						0
	0.0167	1.27				30
	0.0133	1.33				28
	0.0131	1.30				30
	0.0149	1.28				30
	0.0149	1.24				28
						30
						30
						30
						30
		1.28	0.0097			30
		0.02	0.0002			0
						6
	0.0228	1.90				19
	0.0201	1.86				17
	0.0212	1.88				20
	0.0242	1.97				19
	0.0245	1.96				20
						24
						19
						19
						19
		1.91	0.0160			20
		0.04	0.0004			1
						38
	0.0267	2.43				19
	0.0269	2.48				16
	0.0255	2.46				17
	0.0272	2.47				17
						18
						17
						17

T

Determination	Mordanting bath					
	Initial condition			Equilibrial con		
	Potassium dichromate	Hydrochloric acid			Exhaustion	
number	gram of Cr_2O_3 per gram of fabric	normality	pH	pH	percentage	g p f
8						
9						
10						
Mean					41.6	
Deviation						
Percental decrease						
1	none	0.1	--	--		
2						
3						
4						
5						
6						
7						
8						
9						
10						
Mean						
Deviation						
Percental decrease						
1	0.0517	0.1	0.91	0.95		
2						
3						
4						
5						
6						
7						
8						
Mean					49.1	
Deviation						
Percental decrease						

TABLE XVII. (continued)

bath			Fabric				
	Equilibrial condition			Residual weight		As	
	Exhaustion						
	percentage	gram of Cr_2O_3 per gram of fabric	gram	gram	percentage of fabric	gram	pe of
	41.6	0.0302 0.0002					
-			1.7318 1.0408			0.0056 0.0033	
.95			1.0843 1.0679 1.1159 1.1763			0.0309 0.0308 0.0316 0.0335	
	49.1	0.0263 0.0001					

Mordanted fabric						
. weight	Ash		Chromic oxide	Sodium hydroxide 0.2139N	Nitrogen	Breaking strength of wet filling
ercentage fabric	gram	percentage of fabric	gram per gram of fabric	milliliter	percentage of nylon	pounds per inch
						17
						18
						17
		2.46	0.0215			17
		0.02	0.0002			1
						47
	0.0056	0.32				31
	0.0033	0.32				32
						33
						32
						32
						32
						30
						31
						34
						34
		0.32				32
		0.00				1
						0
	0.0309	2.85				17
	0.0308	2.88				17
	0.0316	2.83				14
	0.0335	2.85				17
						12
						17
						16
						17
		2.85	0.0254			16
		0.01	0.0001			1
						50

TA

Determination	Mordanting bath				
	Initial condition			Equilibrial	
	Potassium dichromate	Hydrochloric acid			Exhaustion
number	gram of Cr_2O_3 per gram of fabric	normality	pH	pH	percentage
1		0.125	0.85	--	
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
Mean					50.3
Deviation					
Percental decrease					
1	0.1278	none	4.2	4.8	
2					
3					
4					
5					
6					
7					
8					
9					
10					
Mean					1.25
Deviation					
Percental decrease					

---

[x] Rejected observation.

* The ammonia was distilled into 75.00 ml. of 0.2268N hydrochloric acid

TABLE XVII (continued)

bath			Fabric				
Equilibrial condition				Residual weight		As	
	Exhaustion						
H	percentage	gram of Cr_2O_3 per gram of fabric	gram	gram	percentage of fabric	gram	pe of
--			1.0584			0.0310	
			1.0964			0.0320	
			1.1216			0.0321	
			1.0696			0.0311	
			1.8414	1.8929	102.80	0.0541	
			1.6802	1.7256	102.70	0.0491	
			1.8595	1.9084	102.63	0.0535	
			1.8418	1.8912	102.68	0.0541	
			1.7973	1.8420	102.49	0.0518	
			1.0405				
			1.1027				
			1.0951				
			1.0891				
			1.0623				
	50.3	0.0257 0.0002			102.66 0.08		
4.8			1.0751			0.0051	
			1.0662			0.0049	
			1.0414			0.0049	
			1.0360			0.0049	
	1.25	0.1262 0.0000					

hydrochloric acid.

abric	Mordanted fabric						
	Residual weight		Ash		Chromic oxide	Sodium hydroxide 0.2139N	Nitrogen
ram	gram	percentage of fabric	gram	percentage of fabric	gram per gram of fabric	milliliter*	percentag of nylon
.0584			0.0310	2.93			
.0964			0.0320	2.92			
.1216			0.0321	2.86			
.0696			0.0311	2.91			
.8414	1.8929	102.80	0.0541	2.94			
.6802	1.7256	102.70	0.0491	2.92			
.8595	1.9084	102.63	0.0535	2.88			
.8418	1.8912	102.68	0.0541	2.94			
.7973	1.8420	102.49	0.0518	2.88			
.0405						38.20	11.95^x
.1027						34.99	12.15
.0951						35.44	12.10
.0891						35.60	12.12
.0623						36.79	12.10
		102.66		2.91	0.0260		12.12
		0.08		0.02	0.0002		0.02
.0751			0.0051	0.47			
.0662			0.0049	0.46			
.0414			0.0049	0.47			
.0360			0.0049	0.47			
				0.47	0.0016		
				0.00	0.0000		

Mordanted fabric				
	Chromic oxide	Sodium hydroxide 0.2139N	Nitrogen	Breaking strength of wet filling
centage fabric	gram per gram of fabric	milliliter*	percentage of nylon	pounds per inch
.93				15
.92				13
.86				13
.91				12
.94				13
.92				13
.88				12
.94				14
.88				13
		38.20	11.95^{x}	12
		34.99	12.15	
		35.44	12.10	
		35.60	12.12	
		36.79	12.10	
2.91	0.0260		12.12	13
.02	0.0002		0.02	1
				59
.47				33
0.46				32
0.47				32
0.47				30
				29
				34
				34
				34
				30
				32
0.47	0.0016			32
0.00	0.0000			1
				0

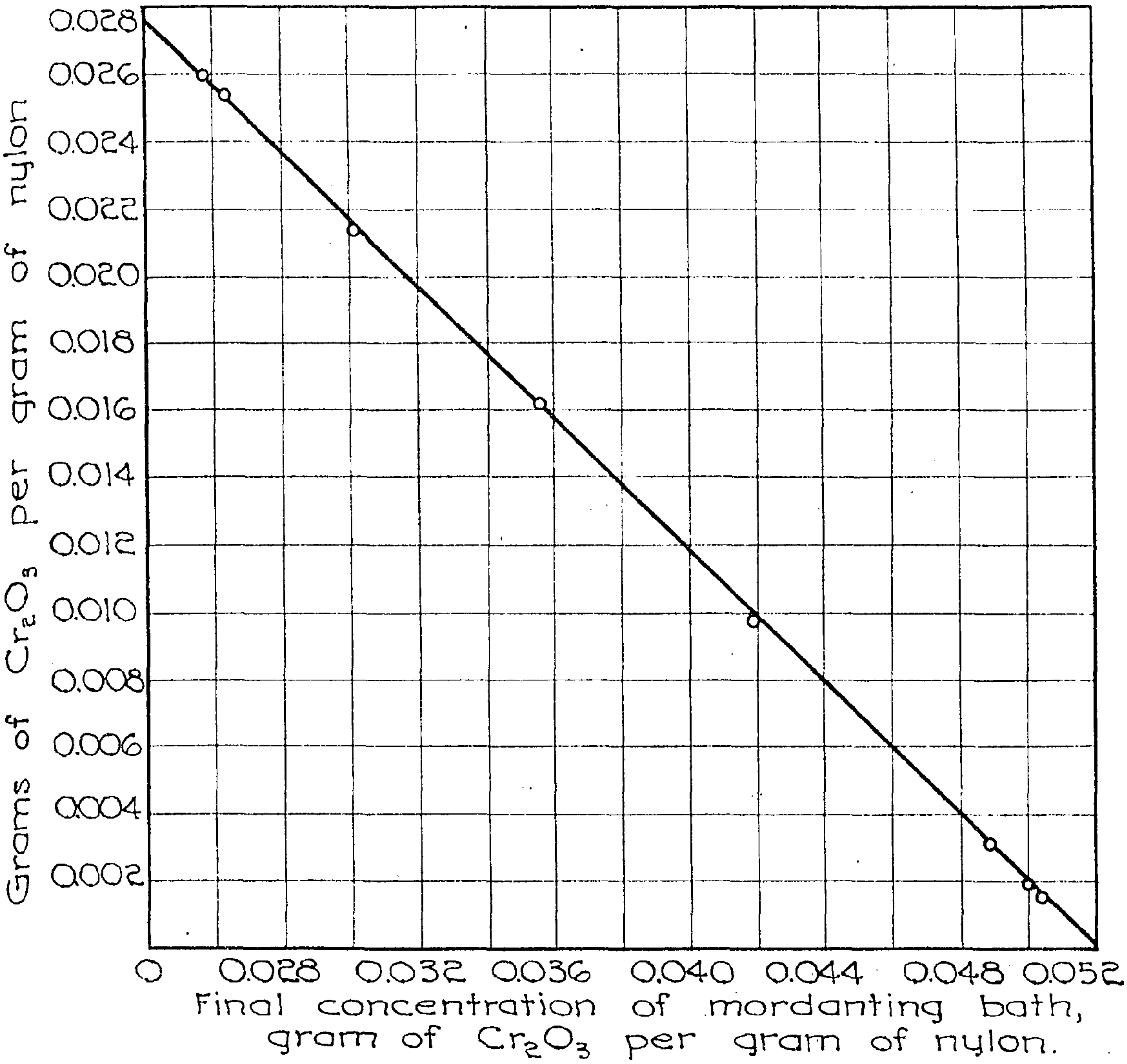

FIG. 3. THE RELATIONSHIP BETWEEN THE FINAL CONCENTRATION OF THE MORDANTING BATH AND THE WEIGHT OF CHROMIC OXIDE FIXED BY NYLON.

Heat

Treatment of breaking-strength specimens with dry heat

Ten specimens were heated in an oven for two hours at 105 to 110°C. or at 134 to 137°C. and then were soaked in distilled water at room temperature for one hour before they were removed singly and broken wet.

In another experiment a General Electric Automatic Iron, featuring "dial the fabric control", was allowed to heat five minutes with the dial switched to each position, "lo", "rayon", "silk", "wool", or "cotton", before being placed on a chromel vs. alumel thermocouple lying on a square of nylon. The temperature over a period of three minutes was obtained from a table (published by Leeds and Northrup) which converts the reading in millivolts on a Leeds and Northrup Potentiometer Indicator to temperature in degrees Centigrade, at a room temperature of 30.5°C. Five breaking-strength specimens which had been ironed two minutes with the thermostat dialed "cotton" were measured for shrinkage and then soaked in distilled water at room temperature for one hour and broken wet.

TABLE XVIII. EFFECT OF HEAT ON THE FABRIC

Determination	Treatment				Breaking strength
	Oven	Ironing			
		Thermostat	Potential	Temperature	
number			millivolt	°C.	pounds per inch
1	two hours at 105-110°C.				29
2					31
3					32
4					31
5					32
6					32
7					30
8					33
9					32
10					30
Mean					31
Deviation					1
1	two hours at 134-137°C.				31
2					30
3					31
4					31
5					32
6					31
7					30
8					30
9					30
10					30
Mean					31
Deviation					1

2	at 105-110°C.				
3					32
4					31
5					32
6					32
7					30
8					33
9					32
10					30
Mean					31
Deviation					1
1	two hours at 134-137°C.				31
2					30
3					31
4					31
5					32
6					31
7					30
8					30
9					30
10					30
Mean					31
Deviation					1
1		"lo"	1.25	31	
2		"rayon"	3.00 to 3.49	74 to 86	
3		"silk"	4.50 to 5.00	110 to 122	
4		"wool"	6.50	159	
5 a*		"cotton"	7.50 to 8.00	184 to 197	34
b					34
c					31
d					34
e					34
Mean					33
Deviation					1

---

* The one-inch strip shrank 6 per cent.

Treatment of fabric with steam

Three glass rods were placed across the top of a four-liter beaker and ten breaking-strength specimens or four one-gram to two-gram samples were tied on the rods with nylon yarns. A watch glass was inverted over the samples and the beaker was set on a tripod in the vertical autoclave of the Biophysical Laboratory. The lid of the autoclave was bolted down and the steam was turned into the chamber. A period of five minutes was allowed to obtain the desired pressure; the pressure was kept at 35, 50, 66, or 77 pounds for five minutes and the steam gradually was allowed to leave the chamber during five minutes. The lid was unbolted during thirty minutes and the specimens were cooled to room temperature, then immersed in distilled water for one hour and broken wet. The one-gram to two-gram samples were rinsed in 100 volumes of distilled water for five three-minute periods, dried at room temperature and used for the determination of residual weight, ash, or nitrogen.

TABLE XIX. EFFECT OF SATURATED STEAM ON THE WEIGHT, AS
TOTAL NITROGEN AND WET STRENGTH OF THE FABRI

Determination	Steam		Fabric of nylon			Ash		Sod hyd 0.2
	Pressure	Temperature		Residual weight				
number	pounds per square inch	°C.	gram	gram	percentage of fabric	gram	percentage of fabric	mil
1*	35	126						
2								
3								
4								
5								
6								
7								
8								
9								
10								
Mean								
Deviation								
Percental decrease								
1	50	138						
2								
3								
4								
5								
6								
7								
8								
9								
Mean								
Deviation								
Percental decrease								

---

* The one-inch strip shrank 6 per cent.

EFFECT OF SATURATED STEAM ON THE WEIGHT, ASH, TOTAL NITROGEN AND WET STRENGTH OF THE FABRIC.

	Fabric of nylon			Ash		Sodium hydroxide 0.2975N	Nitrogen	Breaking strength of wet filling
i-		Residual weight						
	gram	gram	percentage of fabric	gram	percentage of fabric	milliliter	percentage of nylon	pounds per inch
								35
								32
								34
								33
								33
								34
								33
								34
								33
								34
								34
								1
								0
								27
								28
								25
								31
								32
								31
								29
								26
								31
								29
								2
								15

nk 6 per cent.

TABLE XIX. (continued)

Determination	Steam		Fabric of nylon			Ash		Sodium hydrox 0.2975
	Pressure	Temperature		Residual weight				
number	pounds per square inch	°C.	gram	gram	percentage of fabric	gram	percentage of fabric	millilit
1	66	148	1.0705					26.0
2			1.0660					26.3
3			1.0557					27.2
4			1.1120					24.8
5			1.1100	1.1088	99.89	0.0035	0.32	
6			1.0393	1.0373	99.81	0.0033	0.32	
7			1.0976	1.0957	99.83	0.0037	0.34	
8			1.1725	1.1706	99.84	0.0038	0.32	
9								
Mean					99.84		0.32	
Deviation					0.02		0.01	
Percental decrease								
1[c]	77	154						
2								
3								
4								
5								
6								
7								
8								
Mean								
Deviation								
Percental decrease								

---

[x] Rejected observation.

[a] The ammonia was distilled into 75.00 ml. of 0.2268N hydrochloric acid

[c] The one-inch strip shrank 10 per cent.

TABLE XIX. (continued)

—	Fabric of nylon			Ash		Sodium hydroxide 0.2975N	Nitrogen	Breaking strength of wet filling
a-		Residual weight						
.	gram	gram	percentage of fabric	gram	percentage of fabric	milliliter[a]	percentage of nylon	pounds per inch
	1.0705					26.05	12.16	29
	1.0660					26.33	12.10	27
	1.0557					27.21	11.87[x]	23
	1.1120					24.80	12.18	24
	1.1100	1.1088	99.89	0.0035	0.32			25
	1.0393	1.0373	99.81	0.0033	0.32			23
	1.0976	1.0957	99.83	0.0037	0.34			22
	1.1725	1.1706	99.84	0.0038	0.32			29
								25
			99.84		0.32		12.15	25
			0.02		0.01		0.03	2
								26
								24
								19
								22
								24
								20
								23
								24
								20
								22
								2
								35

led into 75.00 ml. of 0.2268N hydrochloric acid
rank 10 per cent.

Light

Chemical treatment of breaking-strength specimens

In all cases, unless otherwise specified, fifty-volume baths were used in treating specimens and ten specimens were treated in each bath. The baths were measured into 250-milliliter Erlenmeyer flasks which were placed in a DeKhotinsky water bath at 25 ± 0.1°C. or 40 ± 0.1°C. and left for one hour to assume equilibrium before the specimens were added. After treatment the specimens were removed, rinsed for five three-minute periods in distilled water and broken wet, or further treated with sodium hydrogen sulfite or ultraviolet light, and then broken wet.

a. With alkali alkyl sulfate

A set of specimens was treated with 0.25 per cent alkali alkyl sulfate for two hours at 25°C.; another set was treated in the same way and exposed to ultra-violet light for 45 hours.

b. With calcium hypochlorite

Ten breaking-strength specimens were treated with: (1) 0.2941N calcium hypochlorite for two hours or six hours at 25°C.; (2) 0.2906N calcium hypochlorite for two hours at 25°C. and then exposed to ultra-violet light for 45 hours; (3) 0.2906N calcium hypochlorite for one hour at 40°C.; or with (4) 0.2875N calcium hypochlorite for two hours at 40°C. or treated in the same way and exposed to ultra-violet light for 45 hours.

c. With formaldehyde

A set of specimens was treated with: (1) 37 per cent or 1 per cent formaldehyde for two hours at 25°C.; or with (2) 1 per cent formaldehyde for two hours at 25°C. and then exposed to ultra-violet light for 45 hours.

d. With iodine

Ten breaking-strength specimens were treated with 0.1000*N* iodine for nine hours at 40°C. and then soaked in 0.3*N* sodium hydrogen sulfite for four hours at 25°C.

e. With phenol

A set of specimens was treated with 1 per cent phenol for two hours at 25°C.

f. With potassium dichromate

Ten specimens were treated with 0.1000*N* potassium dichromate for two hours at 25°C. and then exposed to ultra-violet light for 45 hours.

g. With potassium permanganate

A set of ten specimens was treated with: (1) potassium permanganate 5 or 15 per cent the weight of the fabric, and 0.0 or 0.1*N* as to sulfuric acid or 0.1*N* as to sodium hydroxide, for one hour at 25°C. and then soaked in 0.15*N* sodium hydrogen sulfite for 7.5 hours at 25°C.; or with (2) potassium permanganate 5 or 15 per cent the weight of the fabric, 0.0 or 0.1*N* as to sodium hydroxide, for two hours at 40°C. and then soaked in 0.3*N* sodium hydrogen sulfite for one hour at 25°C.

h. With sodium hydroxide

Ten breaking-strength specimens were treated with: (1) 0.2000N sodium hydroxide for two hours at 25°C. or 40°C., or 0.1000N sodium hydroxide for two hours at 25°C., or treated in each way and then exposed to ultra-violet light for 45 hours; (2) 5 per cent sodium hydroxide for one hour at 100°C. and then exposed to ultra-violet light for 45 hours; or with (3) 10 per cent sodium hydroxide for two hours at 25°C. or treated in the same way and then exposed to ultra-violet light for 45 hours.

i. With sulfuric acid

A set of specimens was treated with 0.1888N or 1.0N sulfuric acid for two hours at 25°C.

Exposure of breaking-strength specimens to ultra-violet light

The light source used was the carbon arc of a Color Fade-ometer, Type C, manufactured by the Atlas Electric Devices Company. The mean temperature of the room was 30°C. The temperature at the face of the fabric was measured with a chromel-alumel thermocouple; the reading of 1.61 m.v. on the Leeds and Northrup Potentiometer Indicator corresponded to a temperature of 40°C. at a room temperature of 32°C. Wet wicks on a rack kept the atmosphere surrounding the samples saturated.

The breaking-strength specimens were attached to the circular rack with Scotch tape. Untreated specimens were exposed to ultra-violet light for 45 hours (over a period of 3.5 days) and sixty hours (over a period of six days), while treated specimens were exposed to ultra-violet light for 45 hours. Then the specimens were removed from the rack, the Scotch tape was cut off, and the specimens were immersed in 100 volumes of distilled water for one hour at room temperature and broken wet.

TABLE XX. EFFECT OF ULTRAVIOLET LIGHT ON THE WET STRENGTH OF THE FABRIC

Determination	Treatment			Exposure to ultraviolet light	Breaking strength of wet filling
	Concentration	Time	Temperature		
number		hour	°C.	hour	pounds per inch
1	none			45	28
2					22
3					21
4					28
5					29
6					29
7					22
8					20
9					25
10					24
Mean					25
Deviation					3
Percental decrease					26
1	none			60	15
2					15
3					17
4					16
5					17
6					24[x]
7					17
8					17
9					17
10					15
Mean					16
Deviation					1
Percental decrease					53

Mean					
Deviation					1
Percental decrease					53
a. Alkali alkyl sulfate					
1	0.25 per cent	2	25	none	34
2					34
3					32
4					37
5					35
6					36
7					37
8					37
9					36
10					37
Mean					36
Deviation					1
Percental increase					6
1	0.25 per cent	2	25	45	17
2					20
3					20
4					17
5					18
6					15
7					17
8					17
Mean					18
Deviation					1
Percental decrease					47
b. Calcium hypochlorite					
1	0.2941N	2	25	none	35
2					35
3					35
4					34
5					33
6					33
7					33
8					30
9					35
10					33
Mean					34
Deviation					1
Percental decrease					0

TABLE XX. (continued)

Determination	Treatment			Exposure to ultra-violet light	Breaking strength of wet filling
	Concentration	Time	Temperature		
number		hour	°C.	hour	pounds per inch
b. Calcium hypochlorite (cont.)					
1	0.2941N	6	25	none	31
2					31
3					32
4					30
5					32
6					32
7					33
8					31
9					32
10					32
Mean					32
Deviation					1
Percental decrease					6
1	0.2906N	1	40	none	32
2					28
3					31
4					32
5					29
6					29
7					31
8					33
9					31
Mean					31
Deviation					1
Percental decrease					9
1	0.2875N	2	40	none	31
2					29
3					29
4					29
5					31
6					29

6					29
7					31
8					33
9					31
Mean					31
Deviation					1
Percental decrease					9
1	0.2875$\underline{N}$	2	40	none	31
2					29
3					29
4					29
5					31
6					29
7					27
8					31
9					25
Mean					29
Deviation					1
Percental decrease					15
1	0.2875$\underline{N}$	2	40	45	16
2					15
3					15
4					14
5					16
6					12
7					16
Mean					15
Deviation					1
Percental decrease					56
1	0.2906$\underline{N}$	2	25	45	23
2					28
3					29
4					25
5					29
6					26
7					30
8					28
9					26
Mean					27
Deviation					2
Percental decrease					20

TABLE XX. (continued)

Determination	Treatment			Exposure to ultra-violet light	Breaking strength of wet filling
	Concentration	Time	Temperature		
number		hour	°C.	hour	pounds per inch
c. Formaldehyde					
1	37 per cent	2	25	none	36
2					35
3					36
4					36
5					35
6					36
7					37
8					35
9					35
10					33
Mean					35
Deviation					1
Percental increase					3
1	1 per cent	2	25	none	35
2					36
3					37
4					36
5					36
6					37
7					33
8					35
9					35
10					37
Mean					36
Deviation					1
Percental increase					6
1	1 per cent	2	25	45	25
2					22
3					22
4					23
5					22

Percental increase					
1	1 per cent	2	25	45	25
2					22
3					22
4					23
5					22
6					18
7					18
8					18
Mean					21
Deviation					2
Percental decrease					38
d. Iodine					
1	0.1000N then 0.30N	9	40		33
2	sodium hydrogen				31
3	sulfite				32
4		4	25	none	29
5					31
6					32
7					31
8					31
9					31
10					29
Mean					31
Deviation					1
Percental decrease					9
e. Phenol					
1	1 per cent	2	25	none	35
2					35
3					33
4					32
5					30
6					29
7					30
8					30
9					28
10					30
Mean					31
Deviation					2
Percental decrease					9

TABLE XX. (continued)

Determination	Treatment			Exposure to ultra-violet light	Breaking strength of wet filling
	Concentration	Time	Temperature		
number		hour	°C.	hour	pounds per inch
f. Potassium dichromate					
1	0.1000N	2	25	45	18
2					18
3					17
4					17
5					17
6					14
7					18
8					20
9					18
Mean					17
Deviation					1
Percental decrease					50
g. Potassium permanganate					
1	50 volumes of distilled water then 0.15N sodium hydrogen sulfite				33
2		1	25		34
3					30
4					32
5		7.5	25	none	30
6					30
Mean					32
Deviation					2
Percental decrease					6
1	5 per cent weight of fabric then 0.15N sodium hydrogen sulfite				26
2					32
3					31
4		1	25		31
5					31
6					31
7		7.5	25	none	31
					26

2	of fabric then 0.15N sodium hydrogen sulfite				32
3					31
4		1	25		31
5					31
6					31
7		7.5	25	none	31
8					26
Mean					30
Deviation					2
Percental decrease					6
1	5 per cent weight of fabric and 0.1N as to sulfuric acid, then 0.15N sodium hydrogen sulfite				24
2					28
3					28
4		1	25		30
5					26
6		7.5	25	none	24
7					30
8					30
9					26
10					27
Mean					27
Deviation					2
Percental decrease					16
1	15 per cent weight of fabric then 0.15N sodium hydrogen sulfite				31
2					30
3		1	25		31
4					31
5		7.5	25	none	32
6					30
7					28
8					24
Mean					30
Deviation					2
Percental decrease					6
1	15 per cent weight of fabric and 0.1N as to sulfuric acid, then 0.15N sodium hydrogen sulfite				22
2					27
3					24
4		1	25		22
5					22
6					26
7		7.5	25	none	27
8					27
Mean					25
Deviation					2
Percental decrease					22

TABLE XX. (continued)

Determination	Treatment			Exposure to ultra-violet light	Breaking strength of wet filling
	Concentration	Time	Temperature		
number		hour	°C.	hour	pounds per inch
g. Potassium permanganate (cont.)					
1	15 per cent weight of fabric and 0.1N as to sulfuric acid, then 0.3N sodium hydrogen sulfite				30
2					28
3					21
4		1	25		20
5					28
6		4	25	none	26
7					24
8					22
9					24
10					24
Mean					25
Deviation					3
Percental decrease					22
1	15 per cent weight of fabric and 0.1N as to sodium hydroxide, then 0.3N sodium hydrogen sulfite				31
2					32
3					29
4		2	25		31
5					30
6		1	25	none	28
7					25
8					29
9					31
10					30
Mean					30
Deviation					1
Percental decrease					6
1	50 volumes of distilled water, then 0.3N sodium hydrogen sulfite				34
2		2	40		30
3					32
4		1	25	none	31

8					[illegible]
9					31
10					30
Mean					30
Deviation					1
Percental decrease					6
1	50 volumes of distilled water, then 0.3N sodium hydrogen sulfite				34
2		2	40		30
3					32
4		1	25	none	31
5					33
6					29
7					33
8					33
9					32
Mean					32
Deviation					1
Percental decrease					6
1	5 per cent weight of fabric, then 0.3N sodium hydrogen sulfite				33
2					34
3		2	40		31
4					34
5		1	25	none	33
6					32
7					34
8					33
9					33
10					31
Mean					33
Deviation					1
Percental increase					3
1	15 per cent weight of fabric, then 0.3N sodium hydrogen sulfite				33
2					32
3		2	40		26
4					31
5		1	25	none	31
6					29
7					26
8					31
9					30
10					27
Mean					30
Deviation					2
Percental decrease					6

TABLE XX. (continued)

Determination number	Treatment: Concentration	Treatment: Time hour	Treatment: Temperature °C.	Exposure to ultra-violet light hour	Breaking strength of wet filling pounds per inch
g. Potassium permanganate (cont.)					
1	15 per cent weight of fabric and 0.1N as to sodium hydroxide, then 0.3N sodium hydrogen sulfite				24
2					29
3					30
4		2	40		24
5					26
6		1	25	none	28
7					28
8					27
Mean					27
Deviation					2
Percental decrease					16
h. Sodium hydroxide					
1	0.2000N	2	25	none	36
2					35
3					35
4					35
5					32
6					35
7					36
8					33
9					33
10					37
Mean					35
Deviation					1
Percental increase					3
1	0.2000N	2	25	45	28
2					29
3					29
4					26
5					25
					30

2					[illegible]
3					29
4					26
5					25
6					30
7					27
8					31
Mean					28
Deviation					2
Percental decrease					18
1	0.1000N	2	25	45	30
2					27
3					27
4					24
5					26
6					28
7					28
8					26
9					27
Mean					27
Deviation					1
Percental decrease					21
1	0.2000N	2	40	none	33
2					30
3					28
4					30
5					34
6					29
7					30
8					33
Mean					31
Deviation					2
Percental decrease					9
1	0.2000N	2	40	45	25
2					28
3					24
4					25
5					27
6					28
7					27
8					25
9					28
Mean					26
Deviation					1
Percental decrease					24

TABLE XX. (continued)

Determination	Treatment			Exposure to ultra-violet light	Breaking strength of wet filling
	Concentration	Time	Temperature		
number		hour	^{o}C.	hour	pounds per inch
h. Sodium hydroxide (cont.)					
1	5 per cent	1	100	45	22
2					22
3					26
4					18
5					22
6					28
7					20
8					15
Mean					22
Deviation					3
Percental decrease					35
1	10 per cent	2	25	none	31
2					35
3					33
4					35
5					33
6					35
7					36
8					33
9					34
10					32
Mean					34
Deviation					1
Percental decrease					0
1	10 per cent	2	25	45	17
2					24
3					17
4					22
5					22
6					20

Mean					[illegible]
Deviation					1
Percental decrease					0
1	10 per cent	2	25	45	17
2					24
3					17
4					22
5					22
6					20
7					22
8					24
9					24
10					24
Mean					22
Deviation					2
Percental decrease					35

i. Sulfuric acid

1	0.1888N	2	25	none	32
2					32
3					33
4					32
5					31
6					32
7					32
8					31
9					31
10					33
Mean					32
Deviation					1
Percental decrease					6
1	1.000N	2	25	none	31
2					31
3					30
4					31
5					32
6					31
7					31
8					32
9					31
10					29
Mean					31
Deviation					1
Percental decrease					9

DISCUSSION OF RESULTS

The analyses of the nylon fabric have been presented in Tables I to XV and are summarized in Tables XXI and XXII. The breaking strength of the fabric in the filling direction was studied in this work because of the high deviation of the warp strength, 4 lb., from the mean, 79 lb., of ten determinations.

The optical properties of the commercial fiber had previously been studied by Frey-Wyssling (108) and Ohara (168). Frey-Wyssling had reported that filaments 19μ in diameter showed very high double refraction, 0.060 as compared to 0.057 for silk and 0.067 for ramie, and interference colors of the third order. The refractive indices of nylon fiber had been reported as $n_{\gamma}D$ 1.580 and $n_{\alpha}D$ 1.520 (34, 39, 108, 166). Ohara (168) had investigated the optical properties of nylon fibers colored with either gold chloride or *Congo Red* and reported that both of these compounds dyed the fiber dichroically in the axial direction. His observations were checked in this laboratory. Nylon was dyed with *Alizarin Blue S*, was mordanted with potassium dichromate, was mordanted and then dyed with *Alizarin Blue S*, or was colored with iodine and then examined microscopically; only in the case of nylon treated with iodine was the fiber colored dichroically.

The melting point of the fiber had been reported as 245°C. (96), 250°C. (74, 98), and 253°C. (125). The melting points observed in this laboratory are in agreement with the mean of the values reported in the literature, 249°C. The

The hygroscopicity of nylon had been reported as less than that of other fibers; the density of the dry fiber had been given as 1.14 and the total regain of moisture as 7.5 to 8.0 per cent (166). At 60 and 70 per cent R. H., the percentage of moisture had been reported as 3.70 and 4.55, respectively (166). The moisture in the fabric at 70 ± 2°F. and 65 ± 2 per cent R. H. in this laboratory has been determined as 2.97 ± 0.04 per cent. The chilliness of the fiber had been attributed to its low hygroscopicity, since less heat is generated by absorption of water (237).

TABLE XXI. ANALYSIS OF FABRIC

Breaking strength of fabric	
Warp direction	
Conditioned, *pounds per inch*	95 (4*)
Wet, *pounds per inch*	79 (4)
Filling direction	
Conditioned, *pounds per inch*	41 (0)
Wet, *pounds per inch*	34 (0)
Distribution of yarns in fabric	
By number	
Warp yarn, *number per inch*	236 (1)
Filling yarn, *number per inch*	104 (0)
By weight	
Warp yarn, *percentage of fabric*	70.04 (0.04)
Filling yarn, *percentage of fabric*	30.16 (0.07)
Elongation of fabric at breaking load	
Warp direction	
Conditioned, *percentage*	32 (2)
Wet, *percentage*	29 (1)
Filling direction	
Conditioned, *percentage*	30 (0)
Wet, *percentage*	31 (1)
Melting point of yarn	
Macroscopic, °C.	248.8 (0.6)
Microscopic, °*C*.	250.5 (0.5)
Moisture, *percentage of conditioned fabric*	2.97 (0.04)
Thickness of fabric, *inch*	0.0035 (0.0001)
Twist of yarn	
Warp yarn, *number per inch, direction*	17 Z (2)
Filling yarn, *number per inch, direction*	15 Z (2)
Weave	2/1 filling rib
Weight of fabric, *ounces per square yard*	2.00 (0.00)
Yarn number	
Warp yarn, *typp*	95.8 (0.3)
Filling yarn, *typp*	97.4 (0.3)

* Deviations are given in parentheses.

Hosino (128) had hydrolyzed the fiber of commercial nylon and obtained adipic acid and hexamethylene diamine. By micro methods Kautz (199) had determined the percental composition of the fiber in nylon hosiery; the values obtained by macro methods in this laboratory compare favorably with Kautz's values (Table XXII). For four determinations by the Kjeldahl method Sakurada and Hizawa (184) had obtained a mean of 11.71 ± 0.23

TABLE XXII. CHEMICAL ANALYSIS OF FABRIC

Determination	Data from this laboratory, macro methods	Data from Kautz, micro methods	Theoretical value
Ash, <u>percentage of fabric</u>	0.35 (0.00*)	0.32	
Carbon, <u>percentage of nylon</u>	63.56 (0.07)	63.43	63.68
Hydrogen, <u>percentage of nylon</u>	9.75 (0.05)	9.85	9.80
Nitrogen, <u>percentage of nylon</u>	12.08 (0.03)	12.52	12.38
Sulfur	none		
Titanium dioxide, <u>percentage of fabric</u>	0.28 (0.01)		

* Deviations are within parentheses.

per cent of nitrogen in the fiber from nylon hosiery as compared to 12.08 ± 0.03 per cent of nitrogen obtained for the woven nylon by the same method in this laboratory. These values for the percental composition of this commercial nylon yield an empirical formula of $C_{12}H_{22}O_2N_2$ for the product obtained by the condensation of adipic acid with hexamethylene diamine.

Determination of the amino nitrogen of nylon by the Van Slyke method (221, 222) did not prove feasible because 30 per cent nitrous acid apparently both hydrolyzed the amide linkages and reacted with these solvents for nylon, *o*-cresol, *tert*-butyl-*m*-cresol, formic acid, and phenol. And none of these substances proved to dissolve nylon appreciably at room temperature: caprylic alcohol, chlorobenzene, *α*-chloronaphthalene, cyclohexanol, dichloropropane, dimethylaniline, dioxane, epichlorohydrin, ethyl caproate, ethyl malonic acid, isobutyl benzoate, isovaleric acid, oleic acid, pyridine, quinoline, tetrahydronaphthalene, toluene, trichlorethane, and a mixture of succinic acid and acetic acid.

British Patent 504,714 describes the delustering of a fibrous superpolyamide with a gas, liquid, or solid or by means of abrasion. From 0.1 to 5.0 per cent by weight of a compound of barium, zinc, titanium, or magnesium or an oil, wax, or resin is recommended (240, French Patent 827,798, U. S. Patent 2,205,722). British Patent 543,291 recommends weighting nylons with tin phosphate and formic acid or phenol. The delustrant of this fabric has been shown to be titanium dioxide, present to the extent of 0.28 per cent.

The effect of fifty-volume baths of aluminum sulfate, containing 0.40, 0.50, 1.50, 3.00, or 6.00 g. of aluminum oxide per gram of fabric, for one hour at 100°C. on the total ash of the fabric is presented in Table XVI. The fixation of aluminum which though slight increased with an increase of pH to 3.00, where a maximum of 0.13 per cent of Al_2O_3 was fixed from a bath containing 0.50 g. of Al_2O_3 per gram of fabric, is in accord with the electrostatic theory of mordanting but suggests that the isoelectric range of this oriented nylon is nearer a pH of 3.00 than a pH of 2.7 (122).

The data in Tables XVII and XXIII show the effect of fifty-volume baths of potassium dichromate in one hour at 100°C. on the weight, chromic oxide, total nitrogen, and wet strength of the fabric. As the concentration of potassium dichromate in the mordanting bath was raised from 0.0207 to 0.1278 g. of Cr_2O_3 per gram of fabric, with accompanying change in initial pH from 4.8 to 4.2, the percentage of chromic oxide fixed by the nylon increased just from 0.09 to 0.16 per cent, the wet strength of the fabric was not changed, and the exhaustion of the bath fell from 4.34 to 1.25 per cent.

The initial pH of the mordanting baths, 0.0517 g. of Cr_2O_3 per gram of fabric (that is, 10 per cent of potassium dichromate per weight of fabric), was changed by the addition of acid or alkali. The effect of increasing the pH to 10.8 was slight. As the pH was lowered to 0.85 by the addition of hydrochloric acid, the percentage of Cr_2O_3 fixed by the fiber increased to 2.60 per cent, the exhaustion of the mordanting bath increased to 50.3 per cent, and the wet strength of the fabric decreased to 41 per cent. Figure 3 shows that a straight-line relationship exists between the chromium fixed by nylon and that left in the mordanting bath. There is no change in the slope of this line at the isoelectric region of the fiber nor where the chromium changes from one ionic combination to another. The amount of chromium fixed by the fiber follows neither Freundlich's nor Langmuir's equation for isothermal adsorption and the ratio of the weight of chromium fixed by the fiber to that left in the mordanting bath is not constant. Rose (181) has postulated that dyeing with an acid dye from an acidic bath occurs because "the fiber must be made less repellent by a surface adsorption of hydrogen ion at the $-\overset{O}{C}-NH-$ link in order to allow the colored anion to secure contact with the fiber in which it is to some extent soluble".

The quantitative results in Table XXIII show that the wet strength of nylon is lowered 38 per cent or more in one hour at $100^{\circ}C$. in solutions of potassium dichromate of pH less than 1.5; however, in order to obtain a good dyeing on nylon with <u>Alizarin Blue S</u>, it is necessary to mordant with potassium dichromate of a still lower pH. Although the loss in wet strength for fabric treated with potassium dichromate of pH 0.85 is 59 per cent,

TABLE XXIII. SUMMARY OF EFFECT OF FIFTY-VOLUME B
DICHROMATE IN ONE HOUR AT 100°C. ON
OXIDE, TOTAL NITROGEN AND WET STREN

Mordanting bath						
Initial condition			Equilibrial condition			Residua weight
Potassium dichromate	Hydrochloric acid			Exhaustion		
gram of Cr_2O_3 per gram of fabric	normality	pH	pH	percentage	gram of Cr_2O_3 per gram of fabric	percent age of fabric
---	---	---	---	---	---	100.00
none	none	5.9	6.4	-	-	
0.0207	none	4.8	5.2	4.34	0.0198	
0.0517	none	10.8[a]	11.0	3.29	0.0500	
	none	4.6	5.0	2.71	0.0503	
	0.0010	3.2	4.0	5.61	0.0488	
	0.0250	1.5	1.7	18.8	0.0420	
	0.0500	1.2	1.3	30.9	0.0357	
	0.0750	1.0	1.0	41.6	0.0302	
	0.1000	0.91	0.95	49.1	0.0263	
	0.1250	0.85	-	50.3	0.0257	102.66 (0
0.1278	none	4.2	4.8	1.25	0.1262	

[a] Sodium hydroxide was added until the solution became 0.1N.
* Deviations are within parentheses.

CT OF FIFTY-VOLUME BATHS OF POTASSIUM
NE HOUR AT 100°C. ON THE WEIGHT, CHROMIC
TROGEN AND WET STRENGTH OF THE FABRIC

	Mordanted fabric					
tion	Residual weight	Ash	Chromic oxide	Nitrogen	Breaking strength of wet filling	
of Cr_2O_3 gram abric	percentage of fabric	percentage of fabric	gram of Cr_2O_3 per gram of fabric	percentage of nylon	pounds per inch	percental decrease
--	100.00	0.35 (0.00*)	-	12.08 (0.03)	34 (0)	-
-		0.31 (0.04)	-		32 (1)	-
198		0.40 (0.01)	0.0009		32 (1)	none
500		0.48 (0.01)	0.0017		31 (1)	3
503		0.45 (0.01)	0.0014		31 (1)	3
488		0.60 (0.03)	0.0029		32 (0)	none
420		1.28 (0.02)	0.0097		30 (0)	6
357		1.91 (0.04)	0.0160		20 (1)	38
302		2.46 (0.02)	0.0215		17 (1)	47
263		2.85 (0.01)	0.0254		16 (1)	50
257	102.66 (0.08)	2.91 (0.02)	0.0260	12.12 (0.02)	13 (1)	59
262		0.47 (0.00)	0.0016		32 (1)	none

N.

the fabric retains its original content of nitrogen. This retention of nitrogen is evidence that hydrolysis of the amide linkage, if it occurs, is random. All of the chromium fixed by nylon from a mordanting bath of pH 0.85 is not changed to Cr_2O_3 by heating at 105 to 110°C.; when this fabric is ashed, the value of 2.91 per cent obtained shows that no loss in weight of fabric during treatment occurs.

Whattam (231) and others (68, 100, 165, 209) had reported that they had obtained deep fast colors on nylon with chrome dyes and the afterchrome process but that the high concentration of acid and potassium dichromate (8 per cent of the latter) necessary to produce satisfactory shades made the method impractical. Tisdale (209) had recommended the mordanting of nylon with 1 per cent potassium dichromate, 1 per cent as to sulfuric acid, before dyeing the fabric black with logwood. Farjou (100) had pointed out that copper salts were good but not economical mordants for nylon.

Dry heat for two hours at 134 to 137°C. (Table XVIII) as well as boiling water for one hour (Table XVII) have been shown to bring about a decrease of only 9 per cent in the wet strength of nylon. It had been reported that a 5 per cent loss in strength of the fiber occurred after four months in air at 65°C., that the fiber was resistant to dry heat at 200°C. in the absence of oxygen but subject to some loss in strength at 225°C. under these conditions and to a decided loss in strength at 245°C. (166). No loss in strength of nylon ironed at 205°C. had been reported (166). The wet strength of the fabric has been shown to be unchanged by ironing at temperatures up to 197°C. (Table XVIII), although the fabric became yellow and shrank 6 per cent when ironed at a temperature between 184 and 197°C. For this reason lower temperatures are recommended for ironing nylon; it is also advisable not to sew cotton with nylon thread since higher temperatures are used for ironing cotton.

Nylon had been described as having a high resistance to steam at 100°C. for six days (166) or at 193.5°C. for three hours (50). A slight loss in strength of a nylon fabric treated with steam at 224°C. and a large loss with steam at 240.5°C. had been observed (50). It has been shown, Table XIX and Table XXIV, that fabric subjected to steam at: (a) 138°C. for five minutes loses 15 per cent of its wet strength; (b) 148°C. for five minutes decreases 26 per cent in wet strength but does not lose weight or nitrogen; and (c) 154°C. for five minutes decreases 35 per cent in strength and shrinks 6 per cent. These results are contrary to those reported anonymously (50). Steam at 126°C. for five minutes has been shown without effect on the wet strength of nylon. Treatment with steam at this temperature is important since these conditions are used in the preboarding process. If filaments are not "set" before dyeing, they develop permanent wrinkles (British Patent 542,034).

TABLE XXIV. SUMMARY OF EFFECT OF SATURATED STEAM IN FIVE MINUTES ON THE WEIGHT, ASH, TOTAL NITROGEN, AND WET STRENGTH OF THE FABRIC

Steam		Fabric of nylon				
Pressure	Temperature	Residual weight	Ash	Nitrogen	Breaking strength of wet filling	
pounds per square inch	°C.	percentage of fabric	percentage of fabric	percentage of nylon	pounds per inch	percental decrease
none	none	100.00	0.35 (0.00)	12.08 (0.03)	34 (0*)	----
35	126				34 (1)	none
50	138				29 (2)	15
66	148	99.84 (0.02)	0.32 (0.01)	12.12 (0.03)	25 (2)	26
77	154				22 (2)	35

* Deviations are in parentheses.

The effect of light on nylon had attracted considerable interest. A sheet of nylon 0.002 cm. thick had been reported to absorb light as follows (141, 166):

Absorption percentage	Wave-length Å
80	2350
10	2550
6	3300
5	3400
0	3600

Cleary had compared the effect of ultra-violet light on nylon and on silk (90). When he exposed breaking-strength specimens to light from a cored carbon arc in a standard Fade-ometer at a distance of approximately 16 in. for 65 hours and 131 hours respectively; nylon showed a reduction in strength of 27 and 40 per cent, while silk showed a reduction of but 10 and 15 per cent (90).

Imperial Chemical Industries (British Patent 545,326) had patented the incorporation of N-N'-polymethylene-bis-o-hydroxybenzamide in the polymer in order to improve the resistance of nylon to ultra-violet light, heat, and oxidation. Treatment of nylon with 37 per cent formaldehyde at 25°C. had been reported to have no effect on the tensile strength but to improve the elasticity, to improve the resistance to degradation by light, and to produce water-repellency (35, 50, British Patents 534,698 and 540,576, U. S. Patent 2,177,637). Owens and Scroggie (U. S. Patent 2,201,741) had improved the resistance of a nylon to discoloration with heat or light by treating it with 0.01 to

5 per cent sodium hydroxide.

The effect of ultra-violet light on the wet strength of the fabric has been presented in Table XX and summarized in Table XXV. The original fabric has been shown to suffer a decrease in wet strength of 26 per cent after 45 hours in ultra-violet light and a decrease of 53 per cent after sixty hours in ultra-violet light at a distance of 10.5 in. from the carbon arc.

Upon treatment with alkali alkyl sulfate and exposure to ultra-violet light for 45 hours, the wet strength decreased 47 per cent, although fabric treated with alkali alkyl sulfate alone gained 6 per cent in strength.

Solutions of calcium hypochlorite at 25°C. have been shown to have very little effect on the wet strength of the fabric although fabric treated for two hours at 40°C. with 0.2875$\underline{N}$ calcium hypochlorite lost 15 per cent in strength. Fabric treated at 25°C. and then exposed to ultra-violet light showed a loss of 21 per cent in strength whereas fabric treated at 40°C. and exposed to ultra-violet light decreased 56 per cent in strength. It is evident that treatment with 0.2906$\underline{N}$ calcium hypochlorite for two hours at 25°C. slightly increased the resistance of the fabric to ultra-violet light. Three per cent sodium hypochlorite for two hours at room temperature and 0.2 per cent sodium hypochlorite for sixteen hours had been reported to have no damaging effect on nylon (77, 166, 201, 204, 231). The Mathieson Alkali Works (British Patent 539,566) had patented the bleaching of nylon with a solution of sodium chlorite and sodium hypochlorite, 0.5 to 5.0 g. of available chlorine per liter and pH 3 to 5. U. S. Patent 2,260,367 describes this bleached product as not damaged but more readily dyed.

An increase of 3 to 6 per cent in wet strength has been observed for fabric treated with 37 or 1 per cent formaldehyde, but exposure to ultra-violet light of fabric treated with 1 per cent formaldehyde resulted in a loss of 38 per cent of its wet strength. Yet it had been reported that 37 per cent formaldehyde increased the resistance of nylon to degradation by ultra-violet light (35, 50).

Fabric treated with 0.1000$\underline{N}$ potassium dichromate and then exposed to ultra-violet light for 45 hours has been shown to lose 50 per cent of its wet strength. This factor should be considered in recommending the use of potassium dichromate as a mordant for nylon.

Treatment of nylon with 3 to 5 per cent potassium permanganate had been recommended for bleaching this fiber since no damage to the fiber had been observed (50, 77, 201, 204, 231). The data of Table XXV show that aqueous potassium permanganate, 5 to 15 per cent the weight of the fabric, at 25°C. or 40°C. has little effect on its wet strength but that solutions of permanganate containing added acid or alkali effected a loss of 16 to 22 per cent. Treatment of nylon with 0.15$\underline{N}$ or 0.3$\underline{N}$ sodium hydrogen sulfite for 7.5 hours or one hour at 25°C. effected a loss of but 6 per cent in its wet strength.

Stott had reported that nylon was very resistant to alkali, withstanding 10 per cent sodium hydroxide for ten hours at 80°C. with a loss in strength of but 5 per cent (125, 201). Fabric treated with 0.1000N or 0.2000N sodium hydroxide at 25°C. or 40°C. has been shown to change but little in wet strength; later exposure to ultra-violet light for 45 hours brought about a loss in wet strength of but 18 and 21 per cent as compared with a loss of 26 per cent by untreated fabric. Specimens treated with 5 per cent sodium hydroxide at 100°C. or 10 per cent sodium hydroxide at 25°C. and then exposed to ultra-violet light have been shown to lose 35 per cent of their wet strength.

The woven nylon has been shown to be resistant to oxidizing agents at room temperature, to be less resistant to oxidizing agents in the presence of an inorganic acid, to be quite resistant to dry heat but not resistant to saturated steam at a temperature greater than 138°C., and to be degraded appreciably with ultra-violet light.

TABLE XXV. SUMMARY OF EFFECT OF ULTRA-VIOLET LIGHT ON THE WET STRENGTH OF THE FABRIC

Treatment			Exposure to ultra-violet light	Breaking strength of wet filling	
Concentration	Time	Temperature			
	hour	^{o}C.	hour	pounds per inch	percentage of original
none			none	34 (0*)	-
none			45	25 (3)	74
none			60		47**
a. Alkali alkyl sulfate					
0.25 per cent	2	25	none	36 (1)	106
0.25 per cent	2	25	45	18 (1)	53
b. Calcium hypochlorite					
0.2941N	2	25	none	34 (1)	100
0.2941N	6	25	none	32 (1)	94
0.2906N	1	40	none	31 (1)	91
0.2875N	2	40	none	29 (1)	85
0.2875N	2	40	45	15 (1)	44**
0.2906N	2	25	45	27 (2)	79
c. Formaldehyde					
37 per cent	2	25	none	35 (1)	103
1 per cent	2	25	none	36 (1)	106
1 per cent	2	25	45	21 (2)	62
d. Iodine					
0.1000N,	9	40			
then 0.30N sodium hydrogen sulfite	4	25	none	31 (1)	91
e. Phenol					
1 per cent	2	25	none	31 (2)	91

d. Iodine					
0.1000N,	9	40			
then 0.30N sodium hydrogen sulfite	4	25	none	31 (1)	91
e. Phenol					
1 per cent	2	25	none	31 (2)	91
f. Potassium dichromate					
0.1000N	2	25	45	17 (1)	50**
g. Potassium permanganate					
50 volumes of distilled water,	1	25			
then 0.15N sodium hydrogen sulfite	7.5	25	none	32 (2)	94
5 per cent weight of fabric,	1	25			
then 0.15N sodium hydrogen sulfite	7.5	25	none	30 (2)	88
5 per cent weight of fabric and 0.1N as to sulfuric acid,	1	25			
then 0.15N sodium hydrogen sulfite	7.5	25	none	27 (2)	79
15 per cent weight of fabric, then	1	25			
0.15N sodium hydrogen sulfite	7.5	25	none	30 (2)	88
15 per cent weight of fabric and 0.1N as to sulfuric acid, then	1	25			
0.15N sodium hydrogen sulfite	7.5	25	none	25 (2)	74
15 per cent weight of fabric and 0.1N as to sulfuric acid, then	1	25			
0.3N sodium hydrogen sulfite	4	25	none	25 (3)	74

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* Deviations are in parentheses.

** A loss of 50 per cent of its strength is one criterion for the failure of a fabric in use.

TABLE XXV. (continued)

Treatment			Exposure to ultra-violet light	Breaking strength of wet filling	
Concentration	Time hour	Temperature °C.	hour	pounds per inch	percentage of original
g. Potassium permanganate (cont.)					
15 per cent weight of fabric and 0.1N as to sodium hydroxide,	2	25			
then 0.3N sodium hydrogen sulfite	1	25	none	30 (1)	88
50 volumes of distilled water, then	2	40			
0.3N sodium hydrogen sulfite	1	25	none	32 (1)	94
5 per cent weight of fabric, then	2	40			
0.3N sodium hydrogen sulfite	1	25	none	33 (1)	97
15 per cent weight of fabric, then	2	40			
0.3N sodium hydrogen sulfite	1	25	none	30 (2)	88
15 per cent weight of fabric and 0.1N as to sodium hydroxide,	2	40			
then 0.3N sodium hydrogen sulfite	1	25	none	27 (2)	79
h. Sodium hydroxide					
0.2000N	2	25	none	35 (1)	103
0.2000N	2	25	45	28 (2)	82

50 volumes of distilled water, then	2	40			
0.3N sodium hydrogen sulfite	1	25	none	32 (1)	94
5 per cent weight of fabric, then	2	40			
0.3N sodium hydrogen sulfite	1	25	none	33 (1)	97
15 per cent weight of fabric, then	2	40			
0.3N sodium hydrogen sulfite	1	25	none	30 (2)	88
15 per cent weight of fabric and 0.1N as to sodium hydroxide,	2	40			
then 0.3N sodium hydrogen sulfite	1	25	none	27 (2)	79
h. Sodium hydroxide					
0.2000N	2	25	none	35 (1)	103
0.2000N	2	25	45	28 (2)	82
0.1000N	2	25	45	27 (1)	79
0.2000N	2	40	none	31 (2)	91
0.2000N	2	40	45	26 (1)	76
5 per cent (1.3N)	1	100	45	22 (3)	65
10 per cent (2.5N)	2	25	none	34 (1)	100
10 per cent (2.5N)	2	25	45	22 (2)	65
i. Sulfuric acid					
0.1888N	2	25	none	32 (1)	94
1.000N	2	25	none	31 (1)	91

SUMMARY

1. A sulfur-free woven nylon, polyhexamethylene adipamide containing 0.28 per cent of titanium dioxide and yielding 0.35 per cent of ash, has been studied in an attempt to determine quantitatively its resistance to mordanting, heat, and light.

2. The effect of fifty-volume baths of aluminum sulfate, containing 0.40, 0.50, 1.50, 3.00, and 6.00 g. of aluminum oxide per gram of fabric, for one hour at 100°C. on the total ash of the fabric was studied. A maximum of but 0.13 per cent of Al_2O_3 was fixed from a mordanting bath of 0.5 g. of aluminum oxide per gram of nylon at a pH of 3.00. The fixation of aluminum which though slight increased with an increase of pH is in accord with the electrostatic theory of mordanting but the data suggest that the isoelectric range of this oriented nylon is nearer a pH of 3.00 than a pH of 2.7.

The effect of fifty-volume baths of potassium dichromate in one hour at 100°C. on the weight, chromic oxide, total nitrogen, and wet strength of the fabric was investigated. As the concentration of potassium dichromate was raised from 0.0207 to 0.1278 g. of Cr_2O_3 per gram of fabric, the pH of the solution and the percental exhaustion decreased, the amount of ash increased slightly, and the wet strength was not changed. As the pH of a solution containing 0.0517 g. of Cr_2O_3 per gram of fabric was decreased by the addition of acid, the percentage of ash and exhaustion increased, and the wet strength decreased. A straight-line relationship has been shown between the Cr_2O_3 fixed by the fabric and that left in the mordanting bath. No change in slope of this line occurs at the isoelectric region of the fiber nor where the chromium changes from one ionic combination to another. Furthermore, the distribution coefficient is not constant. Fabric treated with potassium dichromate of pH 0.85 lost 59 per cent of its wet strength but retained its original weight and amount of nitrogen. This retention is evidence that hydrolysis of the amide linkage, if it occurs, is random. During one hour at 100°C. in mordanting baths of pH less than 1.5, the fabric lost more than 38 per cent in wet strength.

3. Dry heat for two hours at 134 to 137°C. as well as boiling water for one hour each effected a decrease of but 9 per cent in the wet strength of the fabric. The wet strength was not changed by ironing at temperatures up to 197°C., but the fabric yellowed and shrank 6 per cent when ironed between 184°C. and 197°C.

4. Fabric treated with steam at 138°C. for five minutes decreased 15 per cent in wet strength; that treated with steam at 148°C. showed a loss in wet strength of 26 per cent but no loss in weight or nitrogen; and steam at 154°C. effected a decrease of 35 per cent in wet strength and a shrinkage of 6 per

cent. Again in this instance, if hydrolysis of the amide linkage occurs, it is random.

5. After exposure to ultra-violet light for 45 and for 60 hours, the original fabric decreased 26 and 63 per cent in wet strength. The fabric was treated with alkali alkyl sulfate, calcium hypochlorite, formaldehyde, iodine, phenol, potassium dichromate, potassium permanganate, sodium hydroxide, and sulfuric acid. Fabric treated with alkali alkyl sulfate, calcium hypochlorite, formaldehyde, potassium dichromate, and with sodium hydroxide was also exposed to ultra-violet light for 45 hours. Treatment of the fabric with 0.2906$\underline{N}$ calcium hypochlorite or with 0.1000$\underline{N}$ or 0.2000$\underline{N}$ sodium hydroxide for two hours at 25°C. provided some protection to degradation by ultra-violet light as measured by its wet strength.

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ACKNOWLEDGMENT

The writer wishes to express her appreciation to the Biophysical Laboratory for the use of a *KWSZ Photometer*, a *Cameron pH-Meter*, and a high-pressure autoclave and to Dr. Rachel Edgar for the suggestion of the problem and for her helpful advice throughout the progress of this work.