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EFFECTS OF LAUNDERING VARIABLES ON THE FLAME RETARDANT PROPERTIES OF AN 80/20 BLEND OF SAFYR ACETATE AND POLYESTER

by

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INTRODUCTION

Each year in the United States between 150,000 and 250,000 persons are injured, and between 2,000 and 5,000 persons die from burns caused by ignited textiles (12). The majority of those persons are under five or over 66 years of age. In an attempt to reduce the number of injuries and deaths from burning textiles, Congress passed the Flammable Fabrics Act in 1953.

The Act was broadened in 1967 to include home furnishings and to broaden the range of apparel items covered. In 1971, the Federal Trade Commission (FTC) adopted a standard for children's sleepwear. This standard requires that all children's sleepwear sizes 0-6X, after being laundered 50 times, must meet government standards for the vertical flame test. To pass the test, no one single specimen in five replications may 1) burn the entire specimen length of 25.4 centimeters, 2) have a residual flame time of over 10 seconds, and 3) for each five replications the average char length must not exceed 17.8 centimeters (31).

In May 1974, a standard was adopted for children's sleepwear sizes 7-14. This standard requires that all children's sleepwear sizes 7-14 must meet the same requirements for char length as those listed for sizes 0-6X. The requirements concerning residual flame time were dropped (23).

The FTC ruling has been the basis for research on the effects of laundering variables on the flame retardancy of fibers, fabrics and finishes. Pacheco and Carfango (17) found that carbonate-based detergents destroyed the flame-retardant properties of cotton flannelette in 25 launderings or less. Because phosphate detergents are banned in some portions of the country (Dade county, Florida; and the states of New York and Indiana), the majority of the replacement detergents on the market are carbonate-based, or some citrate-based detergents are available.

Perkins (19) found that water hardness also contributed to the loss of flame retardant properties of cotton. Detergent interaction with certain levels of water hardness can cause deposits of calcium and magnesium (referred to as calcium) on the fibers that inhibit their flame retardancy. The extensiveness of the calcium depositions on the fibers can be observed in photoelectronmicrographs of the fabric.

Cotton is ineffective as a flame retardant fabric for children's sleepwear because of the high cost of finishing the fabric and because the finishes are hard to apply either uniformly or permanently. Textile manufacturers are expected to produce fibers and fiber blends that will be effectively flame-retardant for children's sleepwear.

The objectives of this study were to investigate: 1) the effect of laundering variables (detergents, water hardness) on the flame-retardancy of an 80/20 blend of Safyr acetate and polyester fabric, and 2) the amount of calcium deposited on the fabric as a result of the interaction of detergent and water hardness.

One implication of this study is to provide additional information on the launderability of flame-retardant fabrics. The results may be used in further legislation on the flammable fabrics issue and in providing more informative care instructions for the fabrics that are available. This study may also provide a basis for similar studies of different fabrics currently being considered for use in children's sleepwear or for further in depth study of this fabric.

Definition of Terms Used in the Study

Char length: "Char length is the distance from the original lower edge of the specimen exposed to the flame in accordance with the procedure specified in '.4 Test Procedure' to the end of the tear or void in the charred, burned or damaged area" (21).

Children's Sleepwear: "Children's sleepwear means any product of wearing apparel up to and including size 14, such as nightgowns, pajamas or similar or related items such as robes intended to be worn primarily for sleeping or activities related to sleeping. Diapers and underwear are excluded from this definition" (21).

Hard Water: Hard water is water that has calcium and magnesium ions dissolved in it. Hardness can be measured in parts per million (ppm.). Medium hard water has 61-120 ppm.; hard water has 121-180 ppm.; and very hard water has 181 and over ppm. (11).

Residual Flame Time: The residual flame time is defined as the time between removal of the burner from the specimen and the final extinction of molten material or other fragments flaming on the base of the cabinet.

Sample: A sample is a piece of fabric cut from the original bolt. It measures either 29.5 centimeters x 152.4 centimeters or 29.5 centimeters x 101.6 centimeters.

Specimen: A specimen is a unit of fabric (8.9 centimeters x 25.4 centimeters) cut from laundered samples and used to test residual flame time and char length.

REVIEW OF LITERATURE

The following topics were reviewed and studied to provide a background for this project: 1) flame-retardancy and sleepwear standards, 2) flame resistant fabrics and fibers, and 3) laundering effects on flame-retardant fabrics and fibers.

Flame-Retardancy and Sleepwear Standards

The concept of flammability is relative because under the right conditions, anything will burn (8). Only when a fabric is self extinguishable (will not support flame) is it considered flame-retardant.

Miller, Goswami and Turner (14) found two major causes of self extinction: 1) cooling, and 2) decrease of oxygen supply to the fabric. Cooling is involved in the thermal theory of flame-retardancy, which hypothesizes that effective flame-retardants are able to maintain the fabric temperature below the minimum combustion temperature (8). In doing so, energy is either consumed internally or it is conducted away from the flame front. This allows the fabric to cool and the flames to self extinguish.

The gas theory of flame-retardancy involves decreasing the oxygen supply to the fabric, whereby the flame retardant dilutes the flammable gases during combustion or blankets the fabric with an inert atmosphere. In either case, the oxygen supply to the fabric is blocked off and the flames extinguish themselves (8).

The first Flammable Fabrics Act (FFA) was passed in 1953 to ban highly flammable rayon "torch" sweaters. In 1967, the FFA was broadened to 1) cover a wider range of apparel items, 2) give the Secretary of Commerce the right to issue new or revised standards which would protect the public from "unreasonable risk" of death, injury or property damage due to fire, and 3) provide for research into flame injuries and the flammability of fabrics (17).

Studies completed after the FFA was revised in 1967 showed that approximately 150,000--250,000 people were burned each year in fires involving fabrics. Of those burns, 63 percent were caused by clothing ignition and 4,000 people died as a result of their burns. The majority of people burned fatally or seriously in fires involving textiles were under five or over 66 years of age (12).

The results of those studies prompted the Department of Commerce to issue the Children's Sleepwear Standard DOC FF 3-71 on July 29, 1971. The standard required that all children's sleepwear garments, or fabrics intended to be used in children's sleepwear garments, sizes 0-6X, must meet government standards and be labelled that they meet those standards by July 27, 1973. The government standard requires: 1) the char length for any one specimen in five could not be over 25.4 centimeters, 2) the average char length for five specimens must not exceed 17.8 centimeters, and 3) the residual flame time for any one specimen in five must not be greater than 10 seconds (21).

Baum (3) questioned three aspects of the standard: 1) the basis for its adoption, 2) the reasonableness of some of its provisions, and 3) the enforcement policy. Drake (6) mentioned that the studies on burn injuries were not extensive enough or realistic enough to prove the need for flammability standards.

Miller (15) noted that some of the provisions of the flammability standards are unreasonable because of their terminology. He found that too many exact terms were used in either an incorrect or misleading way. Miller also cautioned against the use of char length as a measurement of flame-retardancy because it was difficult to judge whether the fabric was recovering from ignition or whether it was self extinguishing while the char length was forming.

Both Miller (15) and Irvine (12) expressed the opinion that the standard does not provide for burning rates of various fabrics. Baum (3) believed that the high wash temperatures as well as the lack of specified levels of water hardness are unreasonable and that the term "unreasonable risk" was nebulous because it could be used to fit anyone's needs. According to Baum (3, 1), the enforcement policy of the standards is vague. Neither industry nor retailers are given clear cut responsibilities about policy enforcement.

Despite the complaints and disapprovals of DOC FF 3-71, in May 1973 a standard for children's sleepwear sizes 7-14 was proposed by the Department of Commerce. The requirements of the proposed standard differed from DOC FF 3-71 in two ways: 1) the fabrics did not have to be tested in a bone dry state, and 2) the residual flame time did not have to be measured (22). The final notice of the standard for children's sleepwear sizes 7-14 was issued on May 1, 1974. This standard requires bone dry conditioning of the specimens, but it does not require the measurement of residual flame time (23). The new standard will be effective May 1, 1975.

Phase I of the flammability struggle ended for the Consumer Product Safety Commission (CPSC) with the issuance of DOC FF 5-74. In the latter part of 1974, the CPSC plans to do an extensive national survey to determine the frequency and the nature of fire and burn related injuries. The survey will be done on a household to household basis by the National Census Bureau. The CPSC expects that the survey will show the need for additional studies (16).

Flame Resistant Fabrics and Fibers

According to Baum (1) there are three approaches in the manufacturing of flame-retardant fabrics: 1) use of inherently flame-retardant fibers, 2) use of existing fibers modified by addition of non-reactive flame retardants, and 3) application of flame-retardant finishes to fabrics. There are two types of inherently flame-retardant fibers: 1) fibers made from high performance polymers, and 2) fibers made from halogenated polymers.

Fibers from high performance polymers include Nomex and Kynol. Nomex has the wider application, but it has a low moisture regain. Kynol has a high moisture regain, but at the present time it is only available in gold. In addition, both fabrics are too expensive for use in everyday garments (1, 9). Modacrylics, Cordelan, and polyvinyl chlorides are flame-retardant fibers that are made from halogenated polymers. Modacrylic was one of the first flame-retardant fibers and it is one of the least expensive flame-retardant fibers (7). Cordelan is a matrix fiber that is used in flame-retardant thread as well as in fabrics. It is one-third more flame-retardant than modacrylic (1). Polyvinyl chlorides are not used widely because they have poor high temperature characteristics and they also create come dyeing and spinning problems (1). Improved vinyon is available.

Non-reactive flame retardants can be added easily to rayon and acetate because they are both solution spun fibers. Flame-retardant acetate and rayon are both being produced commercially (2, 9). Safyr acetate is produced in both warp and circular knit fabric and is being tested in a woven fabric. The price of Safyr acetate compares favorably with other flame-retardant fibers (2).

Topical finishing is another way of producing flame-retardant fabrics. Pyrovatex and tetrakis(hydroxymethyl)phosphonium chloride (THPC) are currently the leading commercial flame-retardant finishes (2). Fire-Stop cotton is a new flame-retardant finished cotton that shows no significant loss of strength or flame-retardancy after 50 launderings (10). Reeves (20) found that phosphorus containing compounds are effective flame-retardants for cellulosics. Because phosphorus containing compounds do not promote cation exchange, they are effective on cellulosics after repeated home launderings. The quality control of flame-retardant finishes is rigorous. Knits are more difficult to finish than wovens and all finishes are susceptible to changes attributable to laundering (2).

Laundering Effects on Flame-Retardant Fabrics and Fibers

Flame-retardant fabrics may lose their flame-retardant properties either partially or completely when laundered in soap or non-phosphate detergents (5). Since phosphate detergents are banned in some parts of the country, many people have been using soap or carbonate-based detergents for their laundry (5).

Defosse and Carfango (5) tested nine fabrics in their study in which all but one of the fabrics were inherently flame-retardant. Four laundry products were used: 1) phosphate-based detergent, 2) carbonate-based detergent, 3) alkali-built soap, and 4) reduced phosphate-based detergent. Two water hardnesses, 150 ppm. and 300 ppm., were tested for interaction with each laundry product. At both levels of water hardness, all of the fabrics retained their flame-retardant properties after 50 launderings in either a phosphate-based or low phosphate-based detergent, but after 50 launderings in an alkali-built soap at 300 ppm. water hardness, all of the fabrics failed to meet the government standards. Defosse and Carfango (5) found that the adverse effects on flame-retardant properties were due to formation of carbonate salts with the carbonate-based detergent or stearates with the alkali-built soap. As the water hardness increased, so did the deposits found on the fabrics.

Pacheco and Carfango (18) found that flame-retardant additives could be washed out by a carbonate-based detergent or soap. After 50 launderings at 145 ppm. and 300 ppm. water hardness with a carbonate-based detergent, a fabric of acetate and polyester failed to meet the requirements of DOC FF 3-71. The same fabric lost its flame-retardancy after 50 launderings with soap at 300 ppm. water hardness. The tests to determine flammability were conducted according to AATCC test method 124-1969 and DOC FF 3-71. In the same study a cotton flannelette fabric failed to meet the standards of DOC FF 3-71 after 10 launderings with the carbonate-based detergent or 3 launderings with soap. Also, flame-retardancy sometimes could be restored with 10 rewashes in a low phosphate detergent or with one acid sour rinse (18).

Brysson, Piccolo and Walker (4) and Perkins, Drake and Reeves (19) showed that calcium phosphate definitely was deposited on fabrics in laundering because of the interaction of water hardness and detergent. This was true primarily with carbonate-based detergents. LeBlanc and

LeBlanc (13) found that phosphate-based detergents did not lower a fabric's flame-retardancy. They noted, however, that a reduced phosphate-based detergent could cause some calcium deposition because it contained a higher concentration of tripolyphosphate than a regular phosphate-based detergent.

PROCEDURES

The test fabric was a blend of 80 per cent Safyr acetate and 20 per cent polyester. The fabric was knit in a two bar warp tricot construction from 55 denier acetate and 20 denier polyester. The acetate on the fabric surface was brushed to give the fabric a napped appearance.

The fabric was cut into samples, machine laundered 50 times and machine dried. Samples were evaluated at specific points during the 50 launderings.

After the specified number of launderings, samples were cut into specimens and dried 30 minutes in a circulating air oven. Dried specimens were cooled in a dessicator with silica gell dessicant for at least 30 minutes, but not more than 60 minutes. Cooled specimens were evaluated by the vertical flame test and measurement of char length. Photoelectronmicrographs taken by a Poloroid camera attached to a scanning electron microscope were used to show appearance changes of the fibers attributable to calcium deposition.

Sampling Plan

The fabric was cut into samples 29.5 X 152.4 centimeters or 29.5 X 101.6 centimeters for laundering (Appendix A). Ten specimens were evaluated as each 3, 6, and 12 launderings and 15 specimens were evaluated at each 0, 25, and 50 launderings. The ten specimens evaluated at 3, 6, or 12 launderings were cut from the samples 101.6 centimeters in length, and the 15 specimens evaluated at 0, 25, or 50 launderings were cut from samples 152.4 centimeters in length (Appendix B). According to DOC FF 3-71 standards, all of the specimens measured 8.9 centimeters X 25.4 centimeters.

The scraps from the samples were used for the photoelectronmicrographs of the fibers.

Laundering Plan

The fabric was laundered according to AATCC test method 124-1969 under the following conditions:

A. Citrate-based detergent (80 milliliters, 100 grams), water hardness (150 ppm.)

B. Citrate-based detergent (80 milliliters, 100 grams), water hardness (300 ppm.)

C. Carbonate-based detergent (90 grams), water hardness (150 ppm.)

D. Carbonate-based detergent (90 grams), water hardness (300 ppm.)

E. Phosphate-based detergent (90 grams), water hardness (150 ppm.)

F. Phosphate-based detergent (90 grams), water hardness (300 ppm.)

The laundering was done in a Kenmore washer model number 65-14731 using a cycle with a full tub of water, a 14 minute hot water wash (average wash temperature 60.9°C), and a warm water rinse (average rinse temperature 50.9°C). Those conditions came closest to meeting the AATCC test method 124-1969 standards of a 12 minute wash at $60^{\circ} \pm 3^{\circ}C$ and a rinse at $40^{\circ} \pm 3^{\circ}C$. Samples were dried in a Frigidare no vent dryer model DIAF 61 or in a Sears vented dryer model 72086. For each dryer wash and wear settings that met the requirements of the AATCC 124-1969 standards of 60° to 71°C were used.

Because the water coming into the washing machine was softened to 0 ppm., a water hardness concentrate was added to the tub as it was filling. The concentrate was made by adding 441 grams of $CaCl_2.2H_2O$ and 203 grams of $MgCl_2.6H_2O$ to one liter of distilled water. To provide a water hardness of 150 ppm., 25 milliliters of concentrate were added to the wash water, and 50 milliliters were added to provide a hardness of 300 ppm.

Each load of laundry consisted of two samples 152.4 centimeters in length and three samples 101.6 centimeters in length plus an adequate amount of filler cloth (75 X 91 centimeters or 91 X 91 centimeters, 50/50 cotton/polyester sheeting) to make a load of 1.8 kilograms. To maintain the load weight, additional pieces of filler cloth were added as each sample was removed from the load.

Vertical Flame Test

The vertical flame test was done according to the standards set forth in DOC FF 3-71. The machine dried samples were cut into specimens, placed in specimen holders, and dried for 30 minutes in a circulating air oven at $105^{o} \pm 2.8^{o}C$. After removing from the oven, a maximum of five holders were placed in each dessicator for at least 30 minutes, but not more than 60 minutes. After cooling in the dessicator, a specimen was hung in the test chamber. A 3.75 centimeter flame from a Bunsen burner was brought in contact with the lower edge of the fabric for 3.0 ± 0.2 seconds. The residual flame time was measured and recorded. If more than 15 seconds elapsed between the time the specimen was taken from the dessicator and the time the flame was brought in contact with the specimen, the specimen was reconditioned.

When the afterglow of the fabric ceased, the fabric was removed from the specimen holder and creased lengthwise through the highest point of burning. The specimen was then unfolded and 54.4 grams of weight were hooked to the specimen 6.4 millimeters from one lower edge of the specimen. The opposite lower edge was slowly raised until the fabric and weight were suspended in the air. The char length was determined by measuring the total length the fabric had burned and torn.

To pass the standard, the char length of any one specimen may not exceed 25.4 centimeters, and the average char length for five specimens may not exceed 17.8 centimeters. The residual flame time of any one specimen may not be more than 10 seconds.

Microscopic Appearance

The microscopic appearance of the fibers was shown by photoelectronmicrographs taken by a Poloroid camera connected to an ETECH scanning electron microscope at approximately 500-1200X. The photoelectronmicrographs were taken at 0 launderings and at 50 launderings in each condition. If calcium deposits were apparent at 50 launderings, photoelectronmicrographs of samples between 0 and 50 launderings were taken for that specific condition.

FINDINGS

The data were analyzed using a three-way analysis of variance. The results will be discussed according to three major areas: 1) residual flame time, 2) char length, and 3) calcium deposition.

Residual Flame Time

The residual flame time was measured for each of 10 specimens after 3, 6, and 12 launderings and for each of 15 specimens after 0, 25, and 50 launderings with each of the following:

A. citrate-based detergent, 150 ppm.
B. citrate-based detergent, 300 ppm.
C. carbonate-based detergent, 150 ppm.
D. carbonate-based detergent, 300 ppm.
E. phosphate-based detergent, 150 ppm.
F. phosphate-based detergent, 300 ppm.

The fabric failed to meet the government standards set forth in DOC FF 3-71 if one out of five speciments burned over 10 seconds.

After three launderings in a citrate-based detergent, one specimen had a residual flame time of 10.6 seconds. This was the only residual flame time over 10 seconds in 10 specimens, therefore the fabric did not fail to meet the government standards. There were no other individual specimens with residual flame times over 10 seconds when laundered in a citrate-based detergent at either 150 ppm. or 300 ppm. water hardness.

The carbonate-based detergent caused more over-all residual flame time failures than the citrate-based detergent. After twelve launderings at 150 ppm. water hardness, one specimen burned for 16 seconds. Since only one specimen out of ten failed to meet the government standards, the fabric still passed the standard's requirements. A residual flame time of over 10 seconds (10.4, 16.6, 19.4, 10.8, 14.6, 10.8) was obtained after 50 launderings in a carbonate-based detergent at 150 ppm. water hardness with six specimens. Since six specimens had a residual flame time over 10 seconds, the fabric failed to meet the flammability standards after 50 launderings.

A total of seven specimens failed to meet the residual flame time requirements after being laundered in a carbonate-based detergent at 300 ppm. water hardness. After six launderings, the fabric failed to meet government standards; three of the specimens had residual flame times of 13.0 seconds, 19.6 seconds, or 22.1 seconds. The remaining four specimen failures occurred after 50 launderings. The fabric failed to meet the government standards because four out of 15 specimens failed in residual flame time (21.7, 23.7, 24.1, 29.0). The fabric did, however, meet the standards with 12 and 25 launderings.

Of the specimens laundered with a phosphate-based detergent at 150 ppm. water hardness, five had residual flame times of over 10 seconds. One of these failures (16.0 seconds) occurred after 25 launderings and four (18.8, 18.8, 15.0, 16.4) occurred after 50 launderings. With only one failure out of 15 specimens, the fabric still met government requirements after 25 launderings. After 50 launderings, the fabric failed to meet the government standards because four out of 15 specimens had residual flame times of over 10 seconds.

The three-way analysis of variance (Table 1) showed that on an over-all basis, the number of launderings and the type of detergent had a significant effect on the residual flame time. The water hardness did not have a significant over-all effect on the residual flame time, but the interaction of detergent and water hardness did affect the residual flame time significantly. There was also a significant effect on the residual flame time caused by the interaction of detergent and the number of launderings.

Table 1. Condensed Analysis of Variance for Residual Flame Time

Source of variation (RFT)	df	Mean Square	Significance
Number of Launderings	4	114.80	P 0.01*
Type of Detergent	2	81.398	P 0.01*
Water Hardness	1	4.0350	P 0.50 ns
		Second Order	
L X D	8	28.547	P=0.02*
L X H	4	15.185	P=0.29 ns
D X H	2	46.444	P=0.02*
		Third Order	
L X D X H	8	13.758	P=0.35 ns

*significant at 0.05 level   ns--not significant at 0.05 level
RFT--residual flame time

An ordered listing of mean residual flame times attributable to the number of launderings (Table 2) showed that the highest mean (3.32) occurred after 50 launderings and the lowest mean (0.35) after three launderings. The second highest mean (1.16) occurred after six launderings. Although the mean residual flame time was higher after six launderings than it was after 25, 12, or 3 launderings, there were no significant differences between any two of those means. The mean residual flame time after 50 launderings (3.32) was higher (P 0.05) than the mean residual flame times after 25 (0.95), 12 (0.60), or 3 (0.35) launderings.

Table 2. Ordered Residual Flame Time Means for Number of Launderings

Launderings	Mean RFT
50	3.32
	*
6	1.16
	ns
25	0.95
	ns
12	0.60
	ns
3	0.35

*significant at 0.05 level ns--not significant at 0.05 level

A set of ordered mean residual flame times caused by types of detergents (Table 3) showed that a carbonate-based detergent caused the highest residual flame time followed by a citrate-based detergent and then a phosphate-based detergent. The mean residual flame time of the specimens laundered in a carbonate-based detergent (2.20) was higher (P 0.05) than the mean residual flame time of specimens laundered in either a citrate-based (1.06) or phosphate-based detergent (0.57).

Table 3. Ordered Residual Flame Times for Detergents

Detergents	Mean RFT
Carbonate-based	2.20
	*
Citrate-based	1.06
	ns
Phosphate-based	0.57

*significant at 0.05 level ns--not significant at 0.05 level

A significant change in residual flame time when using a carbonate-based detergent and a phosphate-based detergent is associated with water hardness (Table 4). There was no significant change in residual flame time attributable to water hardness when a citrate-based detergent was used.

Table 4. Flame Time Means for the Interaction of Hardness X Detergent

Hardness	Detergents				
	Citrate-based		Carbonate-based		Phosphate-based
150 ppm.	1.33	ns	1.61	ns	1.21
	ns		*		*
300 ppm.	0.78	*	2.80	*	-0.08

*significant at 0.05 level ns--not significant at 0.05 level

With a carbonate-based detergent, the mean residual flame time was higher (P 0.05) when laundered in water with a hardness of 300 ppm. (2.80) than when laundered in water with a hardness of 150 ppm. (1.61). When a phosphate-based detergent was used, the mean residual flame time was lower (P 0.05) in water with a hardness of 300 ppm. (-0.08) than in water with a hardness of 150 ppm. (1.21). Mean residual flame times attributable to detergents were not significantly different at a water hardness of 150 ppm. At 300 ppm. water hardness, the mean residual flame time was lower (P 0.05) when using a phosphate-based detergent (-0.08) than when using a carbonate-based detergent (2.80). The mean residual flame time was also lower (P 0.05) when using a citrate-based detergent (0.78) than when a carbonate-based detergent (2.80) was used. There was no significant difference, however, between the mean residual flame time for a citrate-based and a phosphate-based detergent.

Significant differences in residual flame time occurred with the interaction of detergents and the number of launderings (Table 5). There were no significant differences between the mean residual flame time at any level of laundering when a citrate-based detergent was used. The mean residual flame time after six launderings in a carbonate-based detergent (2.78) was higher (P 0.05) than the mean residual flame time after three launderings in the same detergent (0.38). There were no significant differences between the mean residual flame times after 6, 12, or 25 launderings or between the mean residual flame times after 3, 12, or 25 launderings in a carbonate-based detergent. The mean residual flame time after 50 launderings in the same detergent (5.63) was higher (P 0.05) than the mean at any other tested level. After 50 launderings in a phosphate-based detergent, the mean residual flame time (2.93) was higher (P 0.05) than the mean after 3 (-0.23), 6 (-0.34), 12 (-0.18), or 25 (0.66) launderings. There were no significant differences among the mean residual flame times at these levels.

Table 5. Flame Time Means for the Interaction of Detergents X Launderings

Detergents	Launderings								
	3		6		12		25		50
Citrate-based	0.89	ns	1.03	ns	0.89	ns	1.04	ns	1.41
	ns		ns		ns		ns		*
Carbonate-based	0.38	*	2.78	ns	1.09	ns	1.13	*	5.63
	ns		*		ns		ns		*
Phosphate-based	-0.23	ns	-0.34	ns	-0.18	ns	0.66	*	2.93

*significant at 0.05 level ns--not significant at 0.05 level

After 3, 12, and 25 launderings, there were no significant differences in the residual flame times caused by detergents. However, after six launderings, the mean residual flame time of specimens laundered in a phosphate-based detergent (-0.34) was lower (P 0.05) than the mean residual flame time of those laundered in a carbonate-based detergent (2.78). The mean residual flame time of specimens laundered 50 times in a carbonate-based detergent (5.63) was higher (P 0.05) than the mean residual flame times of those laundered in either a phosphate-based (2.93) or citrate-based detergent (1.41).

<u>Char Length</u>

The char length of each specimen was measured in centimeters. The fabric failed to meet government standards DOC FF 3-71 and DOC FF 5-74 if one out of five specimens had a char length of 25.4 centimeters, or if the average char length for five specimens was greater than 17.8 centimeters. No individual specimens in this study failed to meet the char length requirements specified by the government. At each level of testing, the average char length for each group of five specimens was found to be within the limits set forth in the standards.

A three-way analysis of variance was performed on the char length data (Table 6). The type of detergent used was the only significant (P 0.01) major factor while water hardness X detergent was the only significant (P=0.03) interaction.

The average char length of specimens laundered in a citrate-based detergent (-2.53) was significantly lower (P 0.05) than the average char length of those laundered in either a carbonate-based (-2.04) or phosphate-based detergent (-1.95). There was no significant difference between the average char length of specimens laundered in a carbonate-based (-2.04) or phosphate-based detergent (-1.95) (Table 7).

Table 6. Condensed Analysis of Variance for Char Length

Source of variation (CL)	df	Mean Square	Significance
Number of Launderings	4	3.5776	P≐0.07 ns
Type of Detergent	2	11.499	P 0.01*
Water Hardness	1	2.9157	P≐0.18 ns
		Second Order	
L X D	8	1.5011	P 0.50 ns
L X H	4	1.7468	P≐0.37 ns
D X H	2	6.0050	P≐0.03*
		Third Order	
L X D X H	8	1.6823	P≐0.42 ns

*significant at 0.05 level
CL--char length

ns--not significant at 0.05 level

Table 7. Ordered Mean Char Length for Detergents

Detergents	Mean CL
Citrate-based	-2.53
	*
Carbonate-based	-2.04
	ns
Phosphate-based	-1.95

*significant at 0.05 level
CL--char length

ns--not significant at 0.05 level

The specimens laundered in a carbonate-based detergent with a water hardness of 150 ppm. had a lower (P 0.05) average char length (-2.24) than the specimens laundered in the same detergent with a water hardness of 300 ppm. (-1.65) (Table 8). The average char length of specimens laundered in either citrate-based or phosphate-based detergent did not vary significantly between levels of water hardness.

Table 8. Char Length Means for Interaction of Water Hardness X Detergents

Hardness	Detergents				
	Citrate-based		Carbonate-based		Phosphate-based
150 ppm.	-2.38	ns	-2.24	ns	-2.17
	ns		*		ns
300 ppm.	-2.69	*	-1.65	ns	-1.90

*significant at 0.05 level   ns--not significant at 0.05 level

Calcium Deposition

The amount of calcium deposited on the fabric was evaluated visually using photoelectronmicrographs. The photoelectronmicrographs taken before the fabric was laundered (Plate 1) showed no calcium deposition There were some particles between the fibers that may have been residual particles from the production process. These particles were not abundant and were not attached to the fibers.

Photoelectronmicrographs taken after 50 launderings in a citrate-based detergent at 150 ppm. water hardness showed minimal calcium deposition (Plate 2). Most of the deposits were small, but a few large deposits can be seen. Similar deposits were found in the fabric after it was laundered 50 times in the same detergent at 300 ppm. water hardness (Plate 2).

After 50 launderings in a carbonate-based detergent (Plate 3) at either level of water hardness, the calcium deposits were larger and more prevalent than those seen on the specimens laundered with a citrate- or phosphate-based detergent. The differences in the amount of calcium deposited on the fabric after laundering 50 times in the same detergent, but at different levels of water hardness, was especially evident with the carbonate-based detergent. The deposits were much heavier and the fibers of the specimen laundered 50 times with water 300 ppm. in hardness were almost entirely coated (Plate 3). The heavy coating of calcium was found uniformly throughout the fabric specimen.

Photoelectronmicrographs of the fabric after 50 launderings in a phosphate-based detergent revealed few calcium deposits (Plate 4). The deposits on the fabric were small and scattered. There appeared to be little difference in the amount of deposition on the fabric laundered at a water hardness of 150 ppm. and 300 ppm. The deposition with a phosphate-based detergent appeared similar to deposition with a citrate-based detergent.

Summary

In the United States each year between 150,000 and 200,000 persons are injured and between 2,000 and 5,000 persons die from burns caused by ignited textiles (12). Congress passed a Flammable Fabrics Act in 1953, an amendment to the Flammable Fabrics Act in 1967, and two children's sleepwear standards, DOC FF 3-71 and DOC FF 5-74, in order to reduce the number of injuries caused by burning textiles.

This study was done to investigate the interaction of water hardness and detergent on an 80/20 blend of Safyr acetate and polyester and to investigate the amount of calcium deposited on the fabric.

The fabric was cut into samples and laundered 50 times according to AATCC test method 124-1969. A citrate-based, a carbonate-based, and a phosphate-based detergent were each used at 150 ppm. and 300 ppm. water hardness. The samples were tested for residual flame time and char length after 0, 3, 6, 12, 25, and 50 launderings according to DOC FF 3-71. The calcium deposition was evaluated visually using photoelectronmicrographs.

The fabric failed to meet the residual flame time requirements of DOC FF 3-71 after 50 launderings in a carbonate-based detergent at 150 ppm. and 300 ppm. water hardness. The fabric failed to meet the same standards after six launderings with a carbonate-based detergent at 300 ppm. water hardness and after 50 launderings in a phosphate-based detergent at 150 ppm. water hardness. The failures seemed to be attributable to unknown variables in the testing conditions. The fabric met the government standards for DOC FF 5-74 at all other test conditions. Since the char lengths at all conditions met the government standards, the fabric met the requirements of DOC FF 5-74 at all test conditions.

There was a definite heavy build up of calcium on the acetate fibers at 50 launderings in a carbonate-based detergent with both levels of water hardness. The deposits after 50 launderings in either a citrate-based or phosphate-based detergent were small and scattered. There were no deposits of calcium on the polyester fibers at any of the tested conditions.

PLATE I

PHOTOELECTRONMICROGRAPHS OF SAFYR ACETATE/POLYESTER

Figure 1. Zero Launderings; 580X Magnification

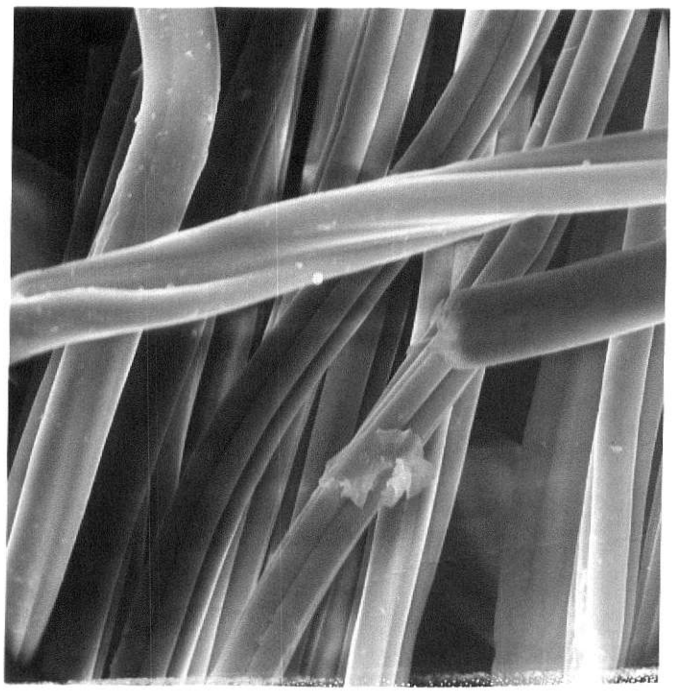

Figure 1

PLATE II

PHOTOELECTRONMICROGRAPHS OF SAFYR ACETATE/POLYESTER

Figure 2. 50 Launderings; Citrate-Based Detergent; 150 ppm.; 740X Magnification

Figure 3. 50 Launderings; Citrate-Based Detergent; 300 ppm.; 380X Magnification

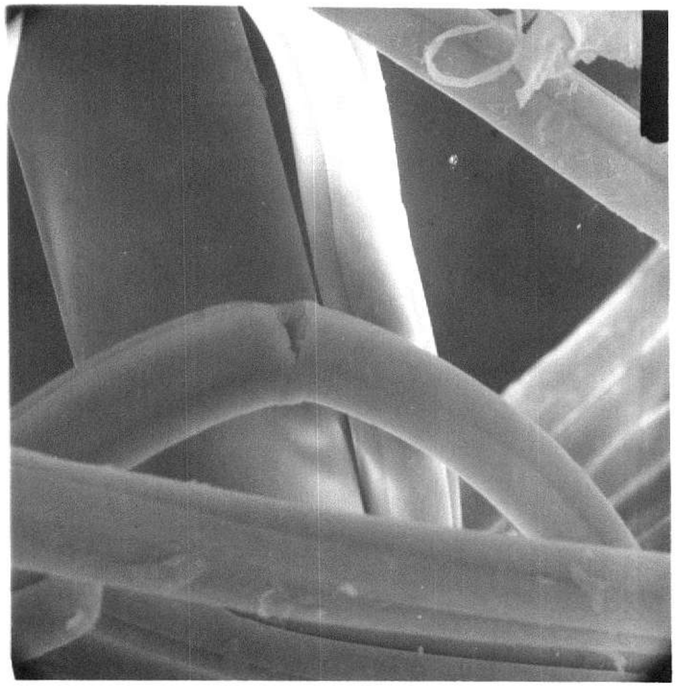

Figure 2

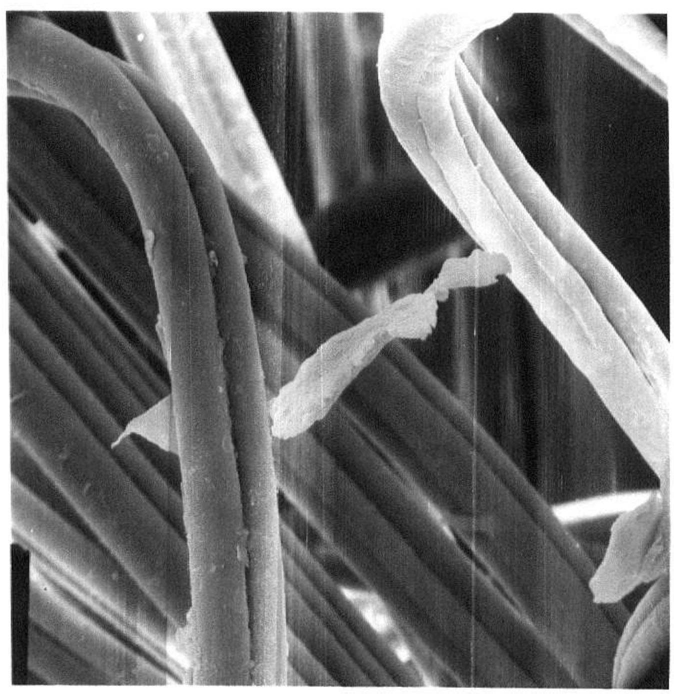

Figure 3

PLATE III

PHOTOELECTRONMICROGRAPHS OF SAFYR ACETATE/POLYESTER

Figure 4. 50 Launderings; Carbonate-Based Detergent; 150 ppm.; 740X Magnification

Figure 5. 50 Launderings; Carbonate-Based Detergent; 300 ppm.; 800X Magnification

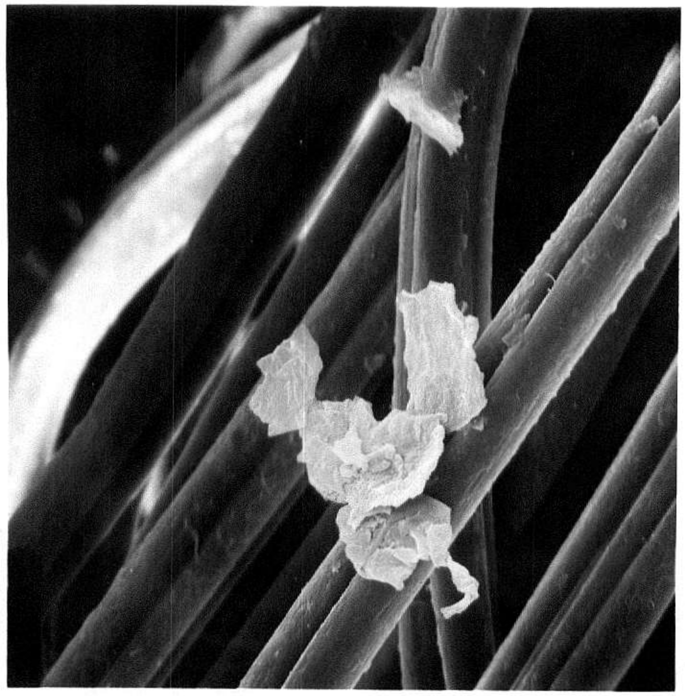

Figure 4

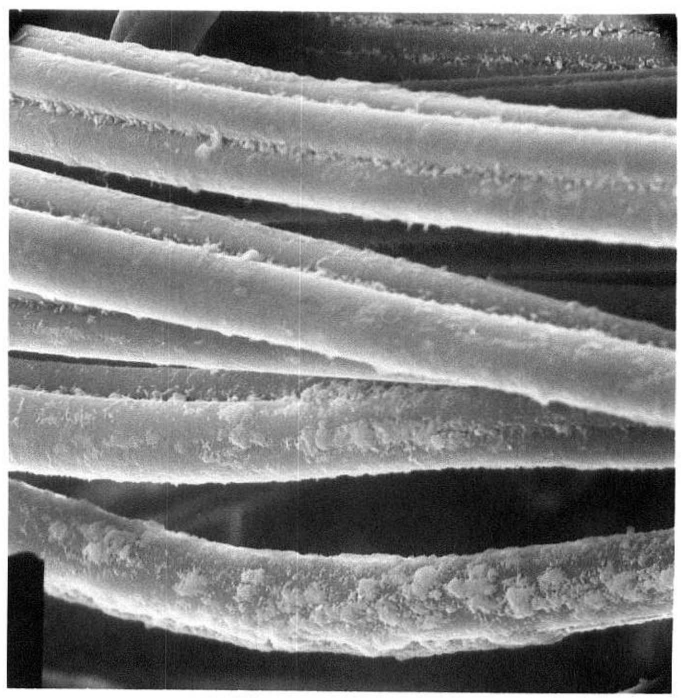

Figure 5

PLATE IV

PHOTOELECTRONMICROGRAPHS OF SAFYR ACETATE/POLYESTER

Figure 6. 50 Launderings; Phosphate-Based Detergent; 150 ppm.; 880X Magnification

Figure 7. 50 Launderings; Phosphate-Based Detergent; 300 ppm.; 720X Magnification

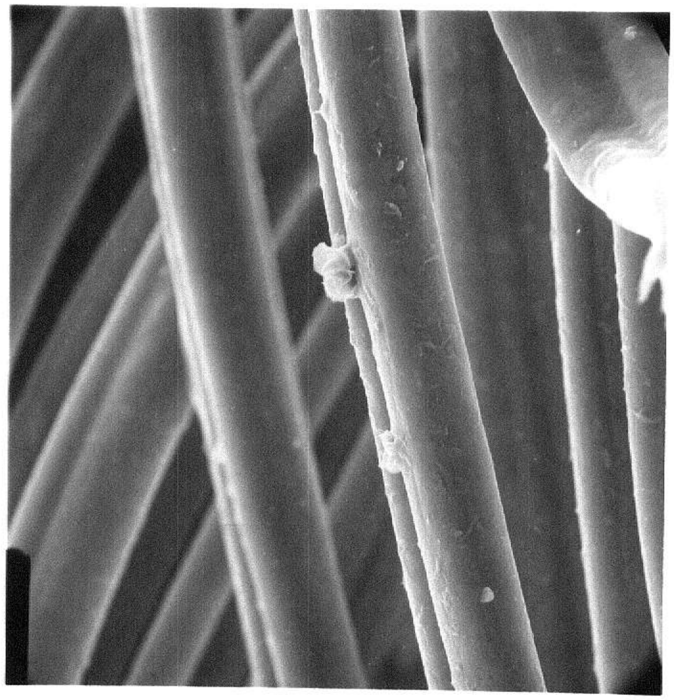

Figure 6

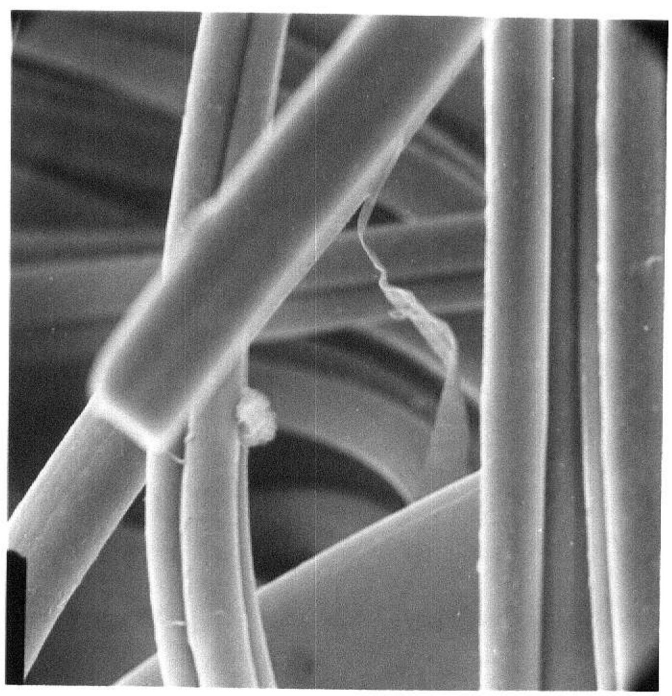

Figure 7

PLATE V

PHOTOELECTRONMICROGRAPHS OF SAFYR ACETATE/POLYESTER

Figure 8. 6 Launderings; Carbonate-Based Detergent; 300 ppm.; 720X Magnification

Figure 9. Comparison of Deposition on Acetate and Polyester; 1120X Magnification

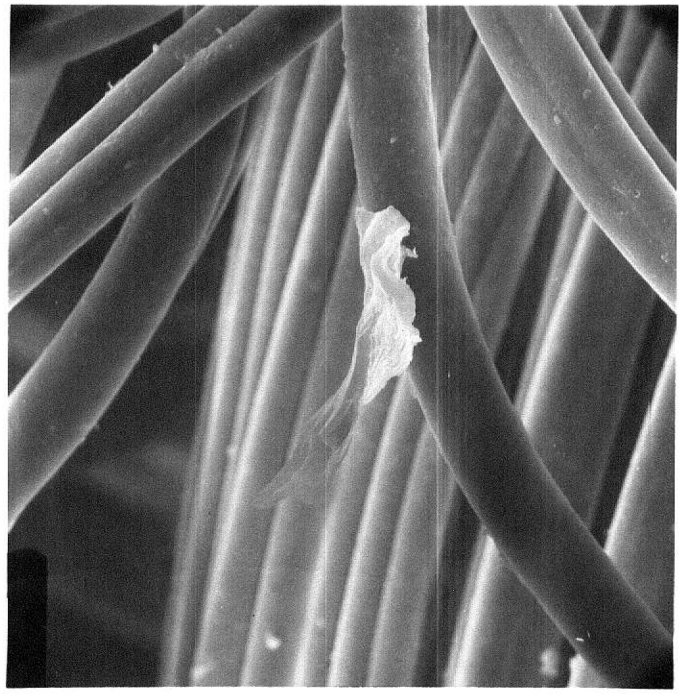

Figure 8

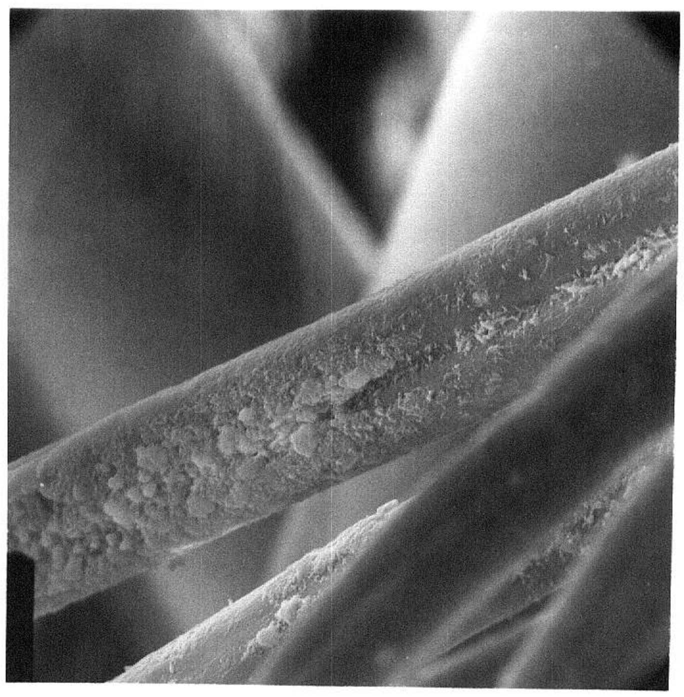

Figure 9

CONCLUSIONS AND RECOMMENDATIONS

An over-all evaluation of the data showed that carbonate-based detergent was the only detergent which had a steady increase in residual flame time with an increasing number of launderings. The char length was not affected by the number of launderings nor were there discernible differences between the citrate-based and phosphate-based detergent.

Statistically, the fabric failed to meet government requirements after six launderings in a carbonate-based detergent at 300 ppm. water hardness. However, this failure appeared to be attributable to causes not readily explainable. The specimens burned quickly to the sides of the specimen holders and continued to flame at the sides for most of the recorded residual flame time. Each of the three specimens that had a residual flame time of over 10 seconds followed this pattern. The photo-electronmicrographs taken of the fabric after six launderings showed little apparent calcium deposition (Plate 5). Since the average char lengths of the specimens laundered six times did not vary appreciably from the average char lengths at any other test level, and the mean residual flame times at 3, 12, and 25 launderings were not significantly different from one another, the results after six launderings seemed to be erroneous. The results, therefore, should not be used in judging the fabric for use in children's sleepwear.

After 50 launderings in a carbonate-based detergent at either level of water hardness, the fabric failed to meet the government standards concerning residual flame time. At both levels of water hardness, the mean residual flame times were extremely higher than the mean residual flame times at any of the other tested levels. The photoelectronmicrographs showed a heavy build up of calcium on the fibers after 50 launderings. The build ups were greater at 300 ppm. water hardness than at 150 ppm. The char length was not affected by the amount of calcium deposition. Even though the build up of calcium was heavy, the acetate fibers were the only fibers affected. No calcium deposits were visible on the polyester fibers throughout the study (Plate 5).

Little calcium deposition was apparent when the fabric had been laundered in a phosphate-based detergent, but after 50 launderings in water 150 ppm. in hardness, the fabric failed to meet the government standards for residual flame time. Again the specimens burned horizontally and supported the flame at the edge of the specimen holder. The char lengths of the specimens laundered in a phosphate-based detergent were not appreciably different from those laundered in a carbonate-based detergent. There were no residual flame time failures among the specimens laundered with a phosphate-based detergent at 300 ppm. water hardness. The char lengths were not large, and the calcium deposition was minimal. The residual flame time failures after 50 launderings at a water hardness of 150 ppm. were probably due to unknown variables in the test conditions.

Of all the detergents tested, the launderings done with a citrate-based detergent yielded the lowest average char lengths and mean residual flame times at both levels of water hardness. A citrate-based detergent seemed to be the best detergent to retain flame-retardant properties of the fabric. A phosphate-based detergent would not affect the fabric much differently than a citrate-based detergent. If the fabric were to be used in children's sleepwear garments sizes 0-6X, the carbonate-based detergent might not be deisrable unless the garment would be laundered less than 25 times during the period in which it was worn. If the fabric were to be used in children's sleepwear garments sizes 7-14, a carbonate-based detergent could safely be used for at least 50 launderings because the residual flame time requirements have been deleted from DOC FF 5-74. Both the citrate- and phosphate-based detergents would be safe for all sizes of garments.

Recommendations

1) In the future, more testing should be done between 25 and 50 launderings. A fabric that passed the requirements of the government standards after 25 launderings, but failed after 50 launderings should be tested at points between 25 and 50 launderings to determine approximately where the failures began to occur.

2) The sampling plan and the number of specimens tested should also be revised to more closely fit the requirements of the government standards.

3) Future testing could also be done with this fabric to show the degree of color loss, strength loss, and shrinkage occurring at each test level.

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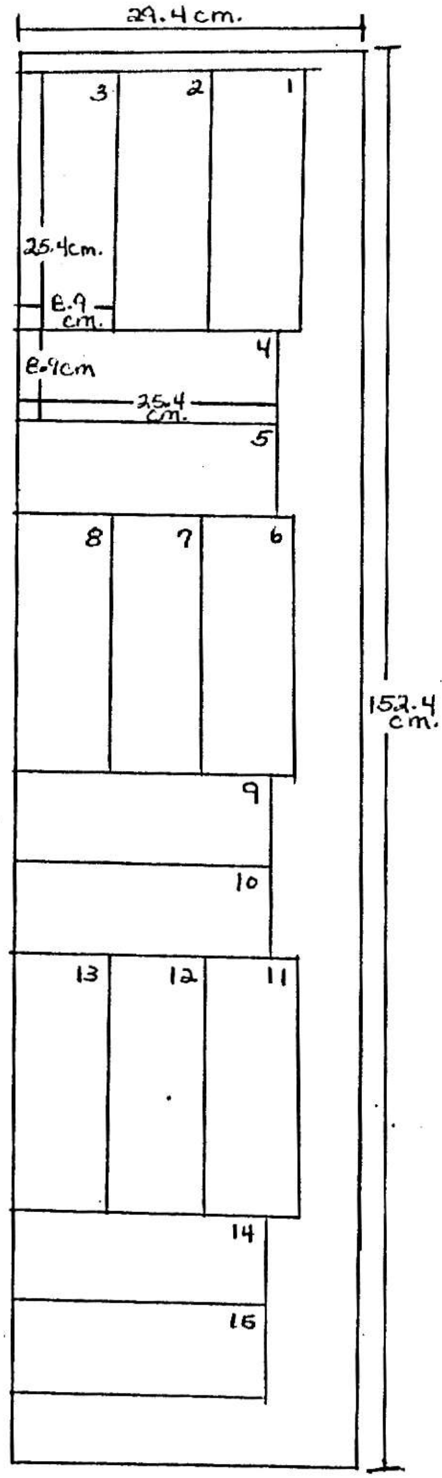
APPENDIX A

LAYOUT FOR CUTTING SAMPLES

THIS BOOK CONTAINS NUMEROUS PAGES WITH DIAGRAMS THAT ARE CROOKED COMPARED TO THE REST OF THE INFORMATION ON THE PAGE.

THIS IS AS RECEIVED FROM CUSTOMER.

APPENDIX A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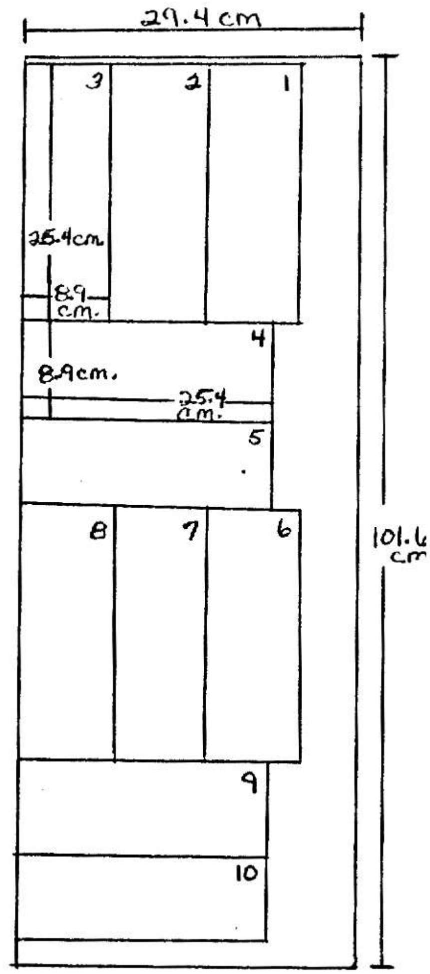
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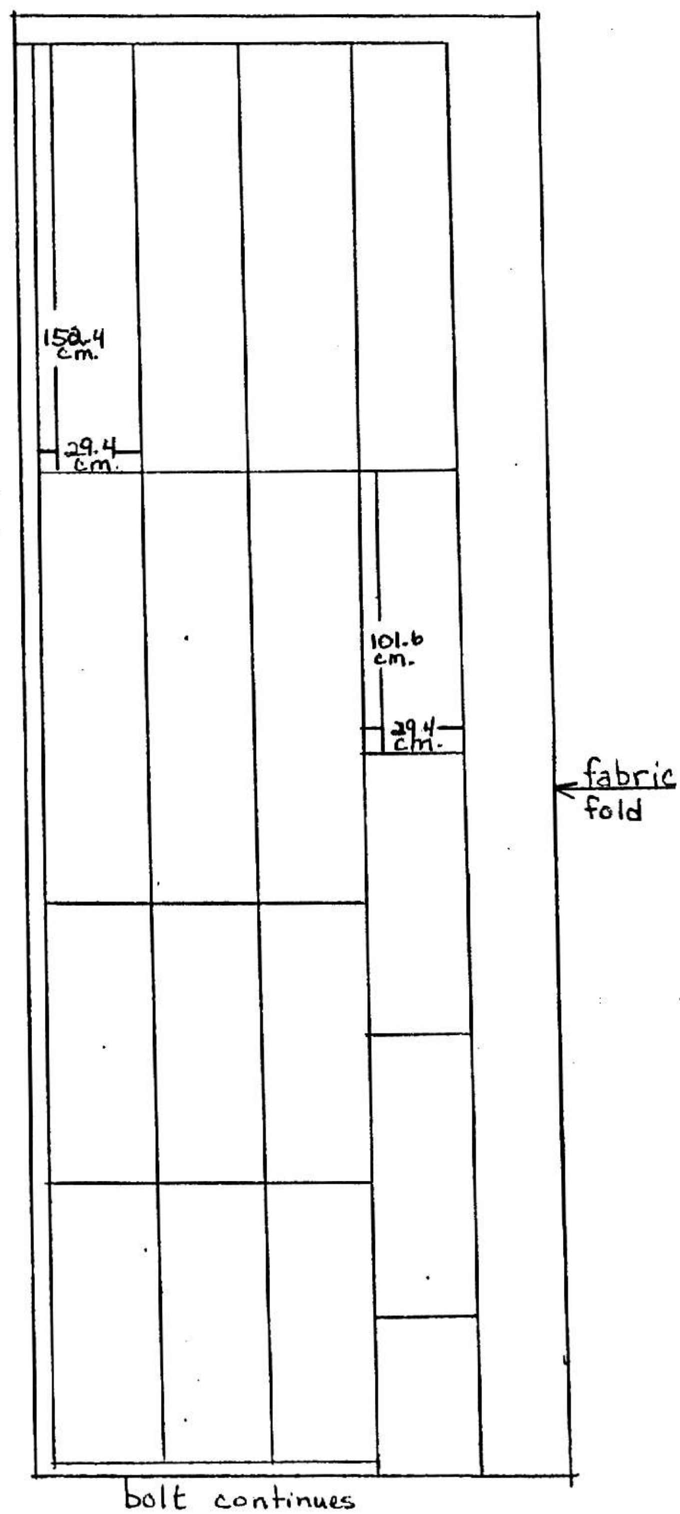
APPENDIX B

LAYOUT FOR CUTTING SPECIMENS

APPENDIX B



EFFECTS OF LAUNDERING VARIABLES ON THE FLAME RETARDANT PROPERTIES OF AN 80/20 BLEND OF SAFYR ACETATE AND POLYESTER

by

KAREN E. KYLLO

B.S., Iowa State University, 1973

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Clothing, Textiles, and Interior Design

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1974

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