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Full Name of Author — Nom complet de l'auteur

DOREEN HUDSON-ROCKLIFF

Date of Birth — Date de naissance 21 · 10 · 40	Country of Birth — Lieu de naissance ENGLAND

Permanent Address — Résidence fixe

17 GLACIER PLACE
ST. ALBERT
ALBERTA
CANADA T8N 1R7

Title of Thesis — Titre de la thèse

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Fire Retardant Finishes for Fiber Art: A Conservation Perspective

by

Doreen Hudson-Rockliff

A THESIS
SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH
IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE
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IN
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YEAR THIS DEGREE GRANTED Fall 1984

(SIGNED) Doreen Hudson-Rockliff

PERMANENT ADDRESS:
17 Glacier Place,
St. Albert, Alberta
Canada T8N 1R7

DATED 23 August 1984

THE UNIVERSITY OF ALBERTA

FACULTY OF GRADUATE STUDIES AND RESEARCH

The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research, for acceptance, a thesis entitled Fire Retardant Finishes for Fiber Art: A Conservation Perspective submitted by Doreen Hudson-Rockliff in partial fulfilment of the requirements for the degree of Master of Science in Clothing and Textiles.

Nancy Kerr
Supervisor

Anne M. Lambert

Karl R. Kopecky

Date 23 August 1984

DEDICATION

To my children:
who probably cannot remember their mother
when she was not a student. Thank you
for making everything worthwhile.

ABSTRACT

Fire Retardant Finishes for Fiber Art:
A Conservation Perspective

by

Doreen Hudson-Rockliff, Master of Science
University of Alberta, 1984

Professor: Dr. Nancy Kerr
Faculty of Home Economics
Division: Clothing and Textiles

Fire safety regulations in Canada usually require that decorative materials in public buildings be capable of passing a vertical flame test either through inherent characteristics or the presence of a fire retardant finish. Fiber art structures are frequently subject to this regulation because of their form and scale.

The purpose of this research was the investigation of three water soluble fire retardant finishes namely, borax:boric acid:diammonium phosphate (7:3:5), X-12, and FlameGard DSH in order to determine what effects they might have on the physical properties of a 100% cotton substrate. X-12 is produced by Spartan Adhesives and Coating Co., Crystal Lake, Illinois and FlameGard DSH is produced by Jersey State Chemical Company/Sybron Corporation, Haledon, N.J. Both compounds are mixtures of inorganic ammonium salts. The amount of add-on necessary for the fabric to pass the vertical flame test was determined. Fabric samples treated with the required level of add-on were subjected to tests to determine changes in dimensions, pH, colour, flexural rigidity and tensile strength. To assess the long term effects of the agents, treated fabrics were subjected to accelerated aging in an Atlas weatherometer with carbon arc lamp. After accelerated aging, tests were conducted to determine changes in pH, colour and tensile strength.

From a conservation point of view all three agents possess some shortcomings. After treatment dimensional change was minimal, pH was acceptable, tensile strength was excellent and there was negligible colour change in the undyed fabrics. Stiffness was considerably reduced at 65% RH but less so at 21% RH. When dyed fabrics were treated all treatments caused a colour change in the red and blue samples. In addition, FlameGard DSH caused noticeable changes in the orange and yellow swatches, borax:boric acid:diammonium phosphate (7:3:5) caused a noticeable change in the orange.

After aging, untreated fabric and that treated with borax:boric acid:diammonium phosphate (7:3:5) became whiter, whilst the fabrics treated with the other two agents turned a tan colour. The pH of all fabrics was lowered, the X-12 treated fabric having the lowest at 3.8 and FlameGard DSH only slightly better at 4.1. Both treatments caused a substantial loss of tensile strength. The borax:boric acid:diammonium phosphate (7:3:5) treated fabric and the control fabric had similar pH levels but the borax:boric acid:diammonium phosphate (7:3:5) treated fabric had a higher loss of tensile strength. The borax:boric acid:diammonium phosphate (7:3:5) treatment leached out at 65% RH and there is some doubt as to whether it survived the accelerated aging process. FlameGard DSH caused corrosion of steel staples with an accompanying strong discolouration of the fabric.

As a result of this research, several recommendations regarding the use of flame retardant finishes on works of art may be made. Investigation of other commercial treatments is recommended. It is also recommended that fiber artists be advised of ways to produce inherently non-flammable structures so that they can avoid the use of finishes. Fiber artists and textile conservators are urged to communicate and cooperate in the production and installation or display of fiber art works in order to ensure that they meet flammability regulations whilst exposing the art work to a minimum of damaging effects. It is suggested that a balance can be achieved between creativity and preservation to ensure that fiber as an art medium has a viable future.

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1. INTRODUCTION

Textile conservation is the means by which any article, composed wholly or in part of textile fibers, is preserved. Conservation is important for all textiles, historical and present day, if they make a contribution to cultural continuity. It is best perceived as a preventive measure to deterioration rather than an interventionist measure after damage has occurred. Preservation is achieved through immediate treatment techniques, long term care procedures and environmental control. The major objectives are avoidance or removal of harmful or disfiguring materials or conditions, the consolidation and preservation of the original textile fibers and the retention of the integrity and aesthetics of the object.

1.1 Statement of the Problem

Fiber art, that is, works of art wholly or in part composed of textile fibers, is being created in increasing numbers and scale. Acceptance as an art medium has made fiber art a much more familiar component of the built environment than it was twenty years ago. Frequently a small percentage of the cost of a new public building is set aside to provide art work for the enhancement of the building. Competition for these large scale commissions is strong and fiber artists must prove themselves worthy of receiving them. The professionalism, or lack of such, displayed by a fiber artist in fulfilling a contract will affect the future of all artists who work in the fiber medium. By successfully dealing with the issue of fire retardancy of their work a major barrier to receiving commissions can be overcome.

Also important to the future of fiber artists and their work is the ability of the art work to remain aesthetically pleasing. Lack of conservation knowledge on the part of fiber artists and owners of fiber art has caused many pieces to be neglected or suffer from a variety of problems such as damage from improper mounting techniques or environmentally induced degradation. Kimura states that "many artists reflect an alarming lack of knowledge of, and interest in, the longevity of their work" (Note 1). Generation of an awareness of the need for conservation of fiber art works is of primary importance so that preventive conservation procedures will be operative from the beginning of the creative process. This awareness should create optimum conditions for long term survival of the art work and obviate the need for costly and intrusive treatment procedures in the future. The topic selected for this study, *Fire Retardant Finishes for Fiber Art*, is just one concern of the textile conservator in the preservation of fiber art.

1.2 Justification

Fiber art has, in artistic terms, matured with the latest generation of artists. Whilst decorative fiber pieces have always been a part of Western culture and have taken many forms, today's individual constructions in fiber have achieved a place and acceptance in the art world unknown twenty years ago. Writing in 1972, in *Beyond Craft: The Art Fabric*, the book that essentially heralded the new art form, Constantine and Larsen introduced their topic thus:

> The Art Fabric is one of the robust, vital arts of our time. Although the development of Art Fabrics is so recent and so varied that they defy classification into the accepted disciplines, it can be claimed with assurance that these are works of art. The artists who create with fiber have united creativity and intuition, principles and skills to form an aesthetic entity. They have molded and extended the meaning of their medium and transcended technique and materials; they have liberated their work from tradition and thus heightened their recognition by critics and public. The great works that have been produced in the fiber medium during the last decade have validated the whole movement. These achievements by a relatively small group of women and men of genius and talent have changed our visual concepts and comprehension. Their works have gained status throughout the world, and while this art form may be in search of nomenclature, it demands and deserves autonomy (p.7).

Fiber arts are now an accepted art form and, as such, as well as being an increasingly familiar component of interior design and decoration they have become gallery pieces in their own right. In Alberta there has been a fourfold increase in the number of commissioned textile works between 1975 and 1980 (Thomas,Note 2). They contribute to the total environment both by their aesthetics and the psychological qualities they impart to the otherwise harsh, cold or sterile environments frequently found in public buildings. Fiber art is one of the art forms that Parkin (1982) says "has the capacity to connect people with the emotive power of the built environment" (p.x).

Traditional techniques have been joined with new ideas and a fresh approach to create fiber pieces that more and more frequently are constructed on a macro or monumental scale. Accompanying the variety and complexity of techniques is an increasing array of materials. Artistic experimentation naturally leads to endless combinations of materials, construction and surface decoration techniques. These "new concepts in designing have initiated...a unique set of problems to be solved" (Davis, 1979, p.24)

The potential for fiber art as a means of fire propagation has been recognized. Concern for public safety has led to increasingly stringent fire codes regulating the use of textile products in public buildings. The National Fire Code of Canada (1980), on which a new Alberta Provincial Code is being based, has set standards of fire resistance for decorative textiles. All decorative textiles are expected to pass specific test procedures before installation can be allowed. If an art work is already installed its removal can be demanded should it fail the test.

Many factors contribute to the potential flammability of fiber art, the major factors being fiber content and construction. Cellulosic fibers, those of vegetable origin such as cotton, linen, jute, sisal and hemp, are the most dangerous due to their organic origin and chemical composition. Where construction is concerned, access and availability of oxygen necessary for continued combustion, is dependent upon the proximity of yarns to one another and the type of yarn construction as well as the nature of the fiber itself. Consequently a tightly constructed fiber piece of heavy rope or cord, having less available oxygen around the individual fibers will be much less flammable than one of lightweight, airy construction or one having a brushed or napped surface.

The interest in fire retardant treatments for textiles is longstanding and there is a wealth of research on treatments for cellulosics. Modern research, however, has focused mainly on the development of durable treatments for clothing and interior furnishing fabrics, that is, treatments which can be applied under large scale, commercial conditions. The focus has generally been on comfort and durability, both of finish and fabric. Whilst these variables are not directly applicable to fiber art, the concepts involved are. Comfort relates to the retention of desirable hand and drape; durability of fabric in wear relates to retention of tensile strength in fibers; and durability of finish to laundering relates to stability of the fire retardant finish to environmental conditions and possibly some cleaning process. The commercial mill application of the finishes, however, limits their usefulness for fiber art.

Other research has focused on the production of inherently non-flammable fibers, that is, fibers with a chemical composition not conducive to combustion. These may help the fiber artist to some extent, but their lack of availability in yarn form, and the restrictions imposed on artistic creativity by limited colour choice or dyeing problems make them only a small part of the available options in meeting fire safety standards. In *The Art Fabric: Mainstream*, Constantine and Larsen, (1981) explain how the necessity of using "non-flammable" materials restricted and altered artist Barbara Shawcroft's original concept for a major fiber piece to be installed in a BART station in San Francisco.

The onus is on the artist to comply with standards. Because of limited application and the specificity of a suitable finish for different fibers fire retardant finishes suitable for fiber art must be found in the commercial reagents available for general textile usage, or be made according to proper specifications from readily available chemicals. Unfortunately the fiber artist cannot relax even when an apparently suitable finish is found. Art works are expected to maintain their original appearance and, unless specifically planned for obsolescence, possess longevity.

Conservation of textile items is becoming increasingly important today because there is now a recognition of the need to preserve textiles of a cultural nature for future generations, for as Pomerantz (1962) says "if continuity in the history of art is important and if we are to continue to learn from and build upon the past we must have more than literary evidence of the existence of a work of art" (p.1). At the same time art textiles are increasingly finding a place in gallery, private and corporate art collections. Generally speaking, collections policies include in their mandate the need for preservation of the art works collected. Nor can the economic factor be denied; many collectors view art as an investment and, as such, textile art cannot be allowed to deteriorate causing loss of value.

The type of care that textile art works will require during their life span is very much influenced by construction and treatment. Preventive conservation measures should begin in the conceptual stages and progress through construction and installation. Given careful thought in the planning and production stages, fiber art works can be produced that will not deteriorate rapidly. When a piece must be treated with a fire retardant finish to comply with fire safety codes, consideration must therefore be given not only to the effectiveness of the fire retardant finish but also to the effect that the finishing treatment may have on the fibers in terms of degradation, colour or dimensional change and change in drape or hand.

A case that occurred recently in the United States exemplifies these problems. The investigation of problems associated with the fire retardant treatment of a wall hanging was the subject of a paper entitled *Preliminary Investigation of a Large Modern Wall Hanging* presented at the 1981 Harpers Ferry Textile Preservation Conference by Karen Clark and Phylis Dillon (Note 3). A very large hanging, forty two and a half feet wide by ten feet deep, forming a wall covering had been treated with a water soluble fire retardant finish which was DuPont X-12 or X-D. Subsequently the reagent had reacted with water vapour in the surrounding air with the result that the chemicals involved had effloresced on the surface of the fabric. The condition had occurred or been aggravated by lack of knowledge of preventive conservation procedures. Proximity to a heating, ventilation and air conditioning (HVAC) area had not been taken into account and no protective barrier had been installed between the wall and the hanging. There were other conservation problems involved in the proposed treatment of the piece but the major problem centered on the need to remove the disfiguring deposit caused by the fire retardant finish.

Forms and styles of the modern fiber art movement have a history of approximately twenty years, but treatment of fiber art works for fire retardancy is sometimes centuries old. Use of such historical agents as ammonium sulfate and borax first used two hundred and fifty years ago is passed on through recipes in publications such as *Fiberarts* and *Shuttle, Spindle and Dyepot* and in handouts to students in fiber arts programs in Alberta. Increasing sophistication of medium and technique in fiber arts needs to be accompanied by an awareness of the conservation of textile materials if a viable future for fiber artists and their work is to be assured. If complying with flammability standards creates too many problems, commissions are going to be more difficult to get, and once received may be more difficult to execute. If a treatment proves damaging to the art work it could also prove damaging to the artist's reputation.

Because cotton has a natural propensity for flammable behavior and is widely used in fiber art work it is the focus of this study. The results obtained are expected to apply to other cellulosic fibers in use.

1.3 Objectives

1.3.1 Major objectives

The purpose of this research study is:

1. To select effective and available fire retardant finishes and evaluate their suitability for use on fiber art.
2. To investigate their short and long term effects on the fiber structure.
3. To make recommendations to artists and textile conservators who are involved in the installation of large scale fiber commissions in public buildings.
4. To provide resource material for textile conservators who may be required to care for a fiber art work subsequent to its treatment with a fire retardant finish.

1.3.2 Minor objectives

It is hoped that the following minor objectives will also be achieved:

1. To generate within the artistic community an awareness of public responsibility for fire retardant treatments for fiber art work.
2. To generate an awareness among fiber artists of conservation concerns for their work.
3. To provide resource material by which fiber artists can learn of the different factors affecting the flammability potential of their art works.
4. To provide the fiber artist with a conceptual framework for understanding the chemical and physical processes related to combustion and fire retardant treatments.
5. To provide the fiber artist with basic knowledge of fire safety standards and standardized test procedures.
6. To provide a basis for discussion of professional and ethical responsibilities for artists and conservators.

1.4 Definition of terms

Terminology in flammability studies is a problem. [illegible] terms such as flame and fire, or resistant and retardant are used interchangeably. [illegible] instances resistant and retardant have been used as a matter of degree. Because there [illegible] consensus in the literature as to exact terminology, flammability terms, together [illegible] considered noteworthy will be used in this study according to these definitions:

add-on - % increase in weight due to absorbed fire retardant agent after treating and drying, based on original dry weight of substrate

afterglow - the process of continued burning, with incandescence, after flame is extinguished

aging - changes undergone in use by a textile material when subjected to potentially destructive agencies other than mechanical, such as light, heat and moisture

combustion - reaction of a substance with oxygen with the release of heat, usually accompanied by flame or glow

conservation - the means by which the true nature of an object is preserved

durable - the ability of a finishing treatment to resist removal by laundering procedures throughout the life of the article

fiber art - constructions produced primarily in the fiber medium conceived for non-utilitarian purposes, individually created by an artist (Davis, 1979)

fireproof - fibers that are essentially unaffected, chemically or physically, by fire; most commonly used examples are glass and asbestos

fire resistant - flame and glow resistant

fire retardant - a substance capable of suppressing, reducing or markedly delaying the process of combustion

flame resistant - will not support a flame after source of ignition has been removed

flammable - capable of burning with a flame

glow resistant - will not continue to burn by the glow mechanism once the source of ignition has been removed or the flame extinguished

ignition - initiation of combustion

inflammable - synonymous with flammable

inherent - belonging to the essential nature of a thing

non-durable - the ability of finishing chemicals to wash or leach out when exposed to laundering or weathering

pyrolysis - the degradation of substances by heat

semi-durable - the ability of a finishing treatment to withstand a limited number of washings

substrate - the material being treated *tendering* - the loss of strength of the substrate as a result of the application of chemical agents

1.5 Delimitations of the study

Because of the unlimited variables inherent in a study of fiber art and fire retardants the limits imposed on the experimental work had to be quite severe. It was decided to restrict the study to three available fire retardant finishes considered viable for a fiber arts application and to apply the finishes to a cotton substrate with a simple fabric geometry. Test methods were selected on the basis of the variables considered most crucial for fiber art.

2. REVIEW OF THE LITERATURE

The focus of this study is the conservation implications of fire retardant treatments for fiber art works composed of cellulosic fibers. Textile conservation as a discipline is relatively new and conservation of contemporary fiber art an even more recent concern, hence there is very little literature which is directly applicable to this topic. The variables involved, that is the flammability of cellulosics, fire retardant finishes for cellulosics, art and conservation, have received a great deal of attention. The review of the literature, therefore, will look at these variables with a view to extracting the information which will directly bear upon the problem being addressed.

2.1 Historical development of fire retardancy

Recognition of the flammability of textile materials and the retardant effect of various topical applications goes back many centuries. The possibility of fires in theatres was particularly great because large quantities of canvas were used for curtains and scenery. These fabrics were the focus of attention as early as 1638 when a mixture of clay and plaster of Paris was recommended as a fire retardant treatment to be incorporated into paint pigments used for painting scenery (Shafizadeh, 1968). A mixture of alum, ferrous sulfate and borax described in British Patent 551 (1735) was the "first noteworthy attempt to flameproof cellulose" (Pitts, 1973, p.134). Ammonium phosphate, still in use today, has a two hundred year old history. In spite of this early start on the fire retardant treatment of textile materials, very little progress was made in the years before World War II towards processes which are not only effective but also free from objectionable effects on fiber appearance and properties. Most early treatments tendered the cellulose fibers, or altered the hand, drape or appearance of the fabrics.

Both the onset of the second World War and an increase in consumer safety concerns led to renewed interest in fire retardancy. In order to eliminate dangerously flammable fabrics from the market, the Flammable Fabrics Act was passed by the United States Congress in 1953. In a 1967 amendment the provisions of the 1953 Act were extended to include household textiles as well as clothing. In addition, the Secretary of Commerce was empowered to issue appropriate standards of flammability for textile articles in commerce in order to protect the public against occurrence of fire leading to death, injury or significant property damage. This amendment generated increased interest in research regarding the chemistry of fire retardants.

Canada became actively involved at the federal level with the passage of the Hazardous Products Act in 1969. This Act led to legislation in 1971 regulating the flammability characteristics of consumer textile products. The early 1970's saw the peak of activity for fire retardancy research. The focus appears to have been almost exclusively on durable, reactive finishes suitable for large scale mill applications under controlled conditions. These finishes have little practical application for fiber art except for those works being created with mill woven cottons. In his review of research in the area of fire retardants for cellulosic textiles, Lyons (1970) states "No other aspect of fire safety has been studied so diligently for so long a time. Yet, at this writing, many problems remain and the need for better methods has never been more acute." (p.165)

The unfortunate 1977 "Tris" incident in the United States, where the fire retardant finish tris(2,3-dibromopropyl) phosphate was found to be a potential carcinogen, and a subsequent revision of flammability standards for children's sleepwear has caused a set-back in research. Chemical companies have withdrawn some fire retardant chemicals from the market due to lack of sales; there has been a decrease in research funds and a marked drop in Patents issued on new fire retardant chemicals (LeBlanc, 1980). Vail, Daigle and Frank (1982) state that "at present the level of fire retardancy research is relatively low and appears to be decreasing" (p.671).

The fire retardant finishes available to the fiber artist today are a selection of old formulations such as borax/boric acid , and proprietary reagents. Many of the old formulations still have practical applicability for fiber arts in terms of fire retardancy; however, the more recent concern of conservation now puts their use into question. Some of the compounds used have been found to have negative effects on fibers causing stiffening, colour change and loss of tensile strength (Drake & Reeves, 1971; Lindsay, 1966; Lyons, 1970). Borax/boric acid treatments have been shown to cause a 28% loss of strength to fabrics at an 11% add-on level (Lyons,1970). A borax:boric acid:diammonium phosphate (7:3:5) preparation has been found to cause stiffening and a rough, undesirable hand. It has also displayed a loss of retardancy properties and a decrease in fabric tensile strength after six months storage at 70 F and 30-50% RH (Little, 1947). Little states that, "the majority of retardants have an appreciable degrading action on cellulose even at normal or slightly elevated temperatures or when subjected to sunlight.

2.2 History of fiber art

In the past many persons considered most textile articles to have a limited lifetime, the exception being decorative items; but even these textiles were frequently perceived as domestic embellishments that would be replaced as fashions changed. To some extent this attitude still prevails. Constantine and Larsen (1981) have clearly shown, however, that there is a growing body of fiber art works that have transcended the fashionable, replaceable, decorative mode to become art in their own right. These new works are deserving of a place in galleries and collections whose mandate includes their long term preservation.

In *The Art Fabric:Mainstream* Constantine and Larsen (1981) present an up to date account of fiber art. In their review of fiber art work from the 1960's period, they describe the art work as having a "basic expression...to do with freedom, revolution and primitive vigor" (p.19). They relate it to the Brutalism appearing in architecture from the same period, using such terms as "heavy, aggressively articulated, rough surfaced, hovering masses" (p.19). Typical materials were organic, naturally coloured, rough fiber, ropes or cords with a heavy elemental focus. Whilst the following decade saw increasing sophistication and experimentation with new techniques "the main attributes of colossal size, organic dynamism, raw expression and intuitive searching did not die" (p.22). This organic expression was frequently achieved with cellulosic fibers such as cotton and flax.

Whereas the majority of the early fiber pieces were created as wall hangings, later works expanded into free standing or suspended, three dimensional sculptural pieces becoming an integral part of interior furnishing as well as interior finishing. In the mid seventies there were changes seen in the choice of materials. There was an increased use of cotton; mill woven cotton fabric became a primary working medium. "Fabric manipulated as a pliable plane became...a major concentration ...the planes sometimes draped on a grandiose scale" (p.52). Cotton fibers have also found their way into the art world through another medium of expression. They are used in the production of handmade paper which is increasing in popularity as an art medium. Traditional methods of painting on canvas must also be included in discussions of fire retardant treatments for cellulosic art works because murals and large scale paintings are being produced that essentially constitute wall finishing treatments.

Such developments in the art world have steered fiber art away from the use of wool, a relatively non-flammable material, and frequently the material of choice for wall hangings, into the realms of the more dangerously flammable cellulosics. The combination of cellulosic materials and monumental scale presents the modern fiber artist with a considerable challenge when the issues of fire retardancy and conservation must be considered.

2.3 Textiles in fires

As "most building fires feed on cellulosic fuels" (Lyons, 1970, p.10) the role of cellulosic textiles in the early stages of the propagation of a building fire has been recognized for a long time. Concern is particularly valid for congregation and egress areas of public buildings. Action can be taken to eliminate some ignition sources. Smoking prohibitions in theatres are an example of regulatory action designed to eliminate accidental fires. Regulations, however, do not compensate for the use of inherently flammable materials. For a safe environment, any textiles used in interiors should not burn readily and "should not constitute fuels in their own right" (Holmes, 1971, p.3).

The intrinsic hazard of a fiber art work is dependent upon not only the materials themselves but also upon the environment they are placed within. According to data collected by the National Fire Incident Reporting System (NFIRS) in 1977 and 1978 textiles were the first-igniter in 21% of cases involving structural fires (Lynn, 1981). As the spread of a fire in a building is dependent upon the surface materials of the walls and ceiling, it can readily be seen that any large scale fiber art piece could be a major propagator of fire. It is the interactions between the complex systems of a building interior that form the hazard rather than the individual components. For example, draperies may ignite easily but have so little heat content that complete combustion causes little damage; upholstered furniture is difficult to ignite but once alight has major fire potential. Together in a geometrical arrangement where the drapes can ignite the furniture they represent a major hazard (Fristrom, 1974).

As well as a means of spreading fire, a fiber art work could also be responsible for the generation of smoke or toxic fumes. These frequently pose a greater hazard to life than flames because they can lead to asphyxiation in a very short time. This problem was graphically illustrated on June 2nd, 1983 when the interior of an Air Canada DC-9 caught fire. The pilot landed the plane safely in Cincinati, but smoke and fumes from burning interior finishings and furnishings killed twenty three passengers before they could escape from the airplane. The complex nature of the fibers and structure of an art work in the fiber medium make it difficult to predict the danger of any piece in the event of ignition. "It is suspected that different toxic gases together constitute a worse physiological hazard than they do separately" (Holmes, 1971, p.3).

2.4 Cellulose structure and combustion

2.4.1 Structure

Cellulose originates as the major component of the cell walls of higher plants. The cellulosic fibers most familiar to fiber artists are cotton, linen (flax), and jute. Pure cellulose is composed of carbon, hydrogen and oxygen, but in nature cellulose varies greatly having a variety of impurities associated w[illegible]. Cotton has been found to be the purest natural form containing more than 90% cellulose; linen is 60 - 85% cellulose (Peters, 1963).

Cellulose is a linear polymer composed of many thousands of anhydroglucose units. Figure 2.1 shows a glucose unit with the carbon atoms numbered. When two glucose units bond together at carbons 1 and 4, one unit rotates 180 degrees and a molecule of water is eliminated. The molecular unit formed is termed cellobiose (Figure 2.2). Cellobiose becomes the repeating unit for the long unbranched chains of the cellulose polymer as successive glucose units are added. The bond formed between the carbon atoms at positions 1 and 4 is an oxygen bridge called the glycosidic link (Peters, 1963). If this bond is broken the molecules depolymerize and all semblance of fiber behavior is lost (Lyons, 1970).

The number of glucose units in the polymer chain is referred to as the degree of polymerization (DP). DP varies a great deal with the different types of cellulosic fibers, and the method of measurement. Native celluloses are reported to have a DP greater than 3000 (Peters, 1963; Shenouda, 1979). When measured by the ultracentrifuge method cotton is said to have a DP of 10,800 and linen a DP of 36,000.

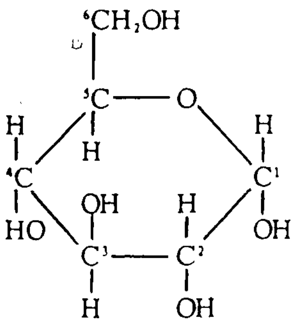


Figure 2.1 A glucose unit with carbon atoms numbered

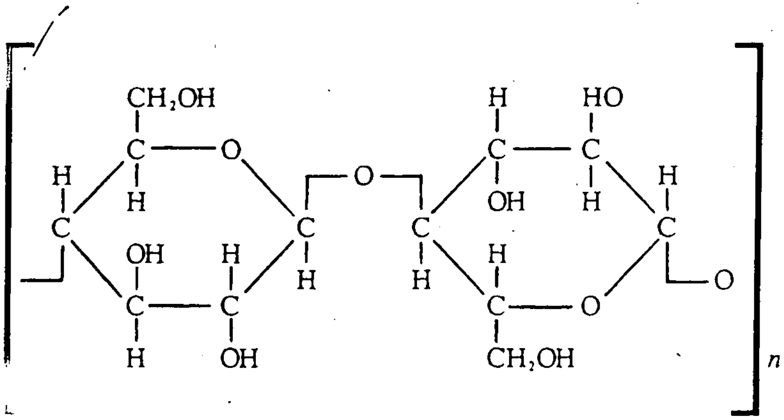


Figure 2.2 Cellobiose

Many of the polymer chains combine together in a parallel linear arrangement to form a cellulose fiber. The chains are bonded together laterally by chemical bonding forces known as hydrogen bonds. These bonds occur between the hydroxyl(OH) groups of adjacent chains. A good explanation of hydrogen bonding theory can be found in Solomons (1976) *Organic Chemistry*. More detailed discussions on the chemical structure of cellulosic fibers can be found in the literature (Lyons, 1970; Peters, 1963; Shenouda, 1979).

2.4.2 Combustion

The combustion of cellulose is an oxidation reaction accompanied by the production of heat and light. It is preceeded by pyrolysis which is the degradation of substances by heat. The products of pyrolysis supply the fuel for the combustion process. Depending upon temperature there are two routes by which thermal degradation can proceed (Peters & Still, 1979). At lower temperatures, 120 - 250 C, degradation is a gradual process of depolymerization, hydrolysis, oxidation, dehydration and decarboxylation leading to loss of strength and a marked reduction in DP. This is essentially the same as the normal aging process.

At temperatures above 250 C, such as would be found in a fire situation, a rapid volatilization occurs producing gaseous and liquid products and charred material (Barker & Hendrix, 1979; Peters & Still, 1979; Shafizadeh, 1968). The larger amount of energy available at higher temperatures results in the breaking of the glycosidic bonds of the main cellulose chain and the hydrogen bonds between the hydroxyl groups leading to loss of fiber structure. Figure 2.3 shows a schematic model of the general reactions involved in the pyrolysis and combustion of cellulose.

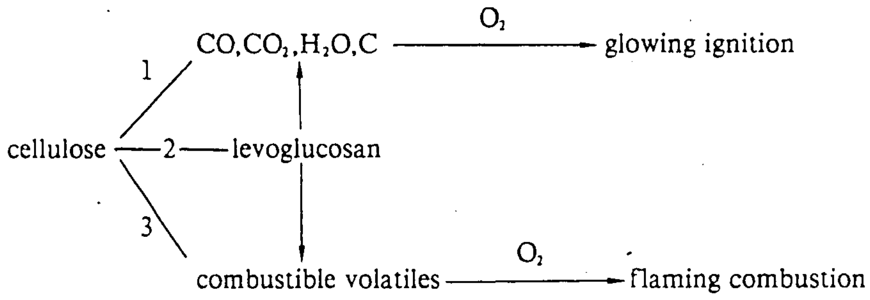


Figure 2.3 The general reactions involved in the pyrolysis and combustion of cellulose (after Shafizadeh, 1968)

The course of the reactions and the decomposition products from the thermal degradation of cellulose are dependent upon the type and rate of heating, the ambient atmosphere and the inorganic impurities and additives in the material. Heating at the lower temperatures favours dehydration and charring reactions (reaction 1 in Figure 2.3). Levoglucosan formation takes place at higher temperatures and is further decomposed at elevated temperatures. The products of high temperature pyrolysis, above 300 C, are many and varied and not all have been identified (Barker & Hendrix, 1979; Lewin & Basch, 1978; Peters & Still, 1979). "The variety and number of the known and unknown decomposition products indicate the extent and complexity of the secondary reactions involved in the pyrolysis and combustion of cellulose" (Shafizadeh, 1968, p.459). Experiments have shown that after a total of 661 minutes of heating cotton cellulose under vacuum at 280 C there was a total weight loss of 70% to volatile fractions composed of tar, water, carbon dioxide and carbon monoxide (Shafizadeh, 1968). The major component of the tar fraction of cellulose pyrolysis has been identified as levoglucosan (Figure 2.4).

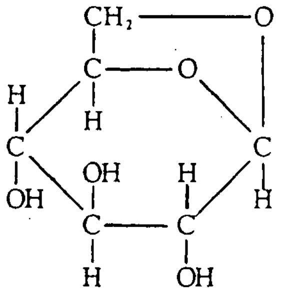


Figure 2.4 Levoglucosan

Extensive discussion of this product can be found in the literature (Barker & Hendrix, 1979; Kilzer, 1971; Lyons, 1970; Peters & Still, 1979; Shafizadeh, 1968).

The depolymerization and dehydration of cellulose to form levoglucosan has been confirmed as the first step in cellulose pyrolysis (Lyons, 1970). Several theories have been advanced to explain the mechanisms by which this reaction is thought to occur. (Shafizadeh, 1968). Levoglucosan is proposed as the key intermediate product implicated in the high flammability of cellulosic fibers (Peters and Still, 1979). A great deal of fire retardancy research focuses on reducing the production of this component of cellulose pyrolysis which can further decompose to flammable volatiles.

The initial thermal degradation of cellulose at temperatures above 250 C provides the necessary combustibles for the combustion process. Ignition, defined as initiation of self-sustaining flaming combustion for an observable time, occurs in the gaseous phase in the presence of oxygen (Miller & Martin, 1978). It may be piloted or spontaneous and it sets in motion the exothermic oxidation reaction accompanied by flame or glow that is combustion. The heat evolved functions in a cyclical manner to continue the pyrolytic process.

Flame and glow are two important aspects of the combustion of cellulose and the question of its retardation. Flaming is the combustion of the volatile products of pyrolysis and glow is the combustion of the carbon residue. Cellulosic fibers can continue to be consumed by glow even when the flame has been extinguished. This property, known as afterglow, is an important factor in the consideration of fire retardant treatments for cellulosics because it is often accompanied by the formation of toxic gases and vapors and can easily start secondary fires (Aenishanslin, 1969). Because flaming and glowing are two separate oxidation reactions, even though they occur concurrently, they "must be considered independently when studying the function of combustion inhibitors" (Little, 1951).

2.5 Factors affecting the combustion of cellulosic textile art works

A fiber art work is a complex structure and, as such, is a "system that reflects not only the material itself but also its geometric configuration and its physical environment" (Rebenfeld, Miller & Martin, 1979). This statement introduces three of the factors that affect the overall flammability potential of an art work in the fiber medium. These are fiber content, density of structure and environmental conditions. A further factor influencing flammability of the structure is the presence of additives or finishing compounds.

2.5.1 Fiber content

This is the major factor to be considered. As previously discussed, cellulose is a highly combustible fiber due to its organic make-up of carbon, hydrogen and oxygen. Also, potential flammability of an art work can be enhanced by the use of combinations of different fibers. In multi-fiber structures interactive behavior occurs; for example, there is a "scaffolding effect" when blends of cellulosic and synthetic fibers are combined in a textile. The flammability of the synthetic fiber is increased because of its close association with the cellulosic fiber. Essentially the thermoplastic fiber is not allowed to drip away as it normally would and the cellulosic fiber or char acts as a wick for the molten polymer (Pintauro, 1978).

2.5.2 Geometric configuration

This aspect of an art work is obviously of major importance in the aesthetic sense, but it also influences the rate at which the textile will burn. Physical manipulation of fibers, yarns and fabrics is the means by which an artist conceptualizes an art piece in the fiber medium. The form it takes is based upon the symbolism the artist wishes to project. It should be recognized, however, that the spatial arrangements of the materials and the compactness of the fibers used will affect the surface area to volume ratio of the textile. The higher the surface area to volume ratio, the greater the air permeability and, therefore, the greater the availability of oxygen to support combustion (Hendrix, Drake & Reeves, 1973). Furthermore, McCullough (1978) found that testing and evaluation of fabrics as single layers may have little relationship to the potential hazards of burning assemblies because it was found that the flammability hazard associated with assemblies was greater than that expected from the sum of the single layers.

2.5.3 Environmental conditions

Shafizadeh (1968) states that the rate of combustion or propagation of a fire is highly sensitive to prevailing environmental conditions. This statement is supported by the fact that environmental conditions have been found to affect the Oxygen Index (OI) values of materials when tested in a laboratory setting (Beninate, Drake & Reeves, 1973; Drake, 1972; Miller, Goswain & Meiser, 1975). "OI is a precise, reproducible measurement of the minimum concentration of oxygen, in a mixture of oxygen and nitrogen, that will just support combustion of a material" (Timpa, Segal & Drake, 1975, p.86). Therefore, the higher the OI level the lower the flammability of the textile. According to Miller et al (1975) "it is generally expected that the presence of moisture in a textile fabric will have some influence on its flammability behavior" (p.328). Their research showed that OI values increased with an increase in moisture content in a complex manner. These results support the findings of Drake (1972) who showed that moisture content significantly affects the flammability characteristics of many fibers. Hendrix, Beninate, Drake and Reeves, (1973) also found that OI values decrease with increasing environmental temperatures; that is, the higher the temperature the higher the flammability potential. Variations found in actual use conditions of a fiber art, in many instances, may not be large enough for the effects of moisture and temperature to play a major role in flammability potential, although this cannot be assumed in a country such as Canada where environmental conditions vary immensely.

2.5.4 Additives and impurities

Because flammability is a function of the chemical composition of the textile, any modification of the cellulose substrate will affect its flammability behavior. Hendrix, Drake & Reeves, (1973) state that sample purity was shown to have a dramatic effect on OI values, impurities causing substantially higher OI values than those obtained from purer samples. The yarns and fabrics of fiber art work may contain such additives as dyes or have been subjected to finishing treatments such as Scotchgard®, a fluorocarbon finish designed to impart water and oil repellancy. Because these are chemical additions to the cellulose substrate they can affect the products of decomposition and the rate and course of combustion reactions. Hilado and Huttlinger (1982) found that direct dyes alter the mode of decomposition of cellulose, and Yeager and Chapin, (1972) state that "dyes themselves can increase the flammability of a fabric [and that] printing pastes can also contribute to increased flammability" (p.39).

Pintauro (1978) states that "the overall flammability potential of a [fiber art work] is based on the behavioral components of textile flammability" (p.2). The parameters given are ease of ignition, burning rate, heat emission and ease of extinguishment. These behavioral parameters are governed by the chemical and physical factors of the preceeding discussion. Welker (1973) indicates that "it is still not possible to predict, without benefit of experimental data, whether or not ignition will occur under a given set of conditions, when ignition will occur, and if burning will be sustained once ignition has occurred" (p.13). Each fiber art construction has different chemical, physical and thermal properties due to the unique nature of art; thus, the complex nature of its flammability is unique.

2.6 Mechanisms of fire retardancy of cellulosic materials

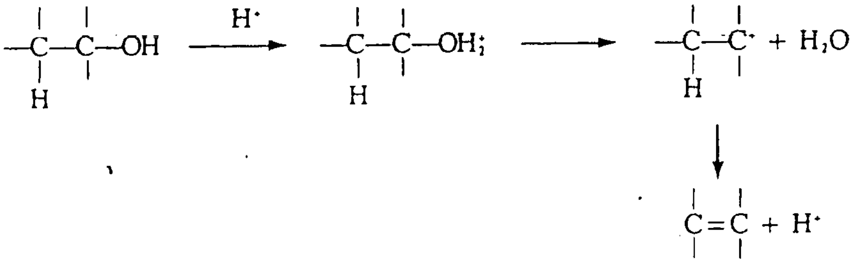
"The theoretical aspect of the mechanism of fabric flame retardancy is complicated both because of the burning process itself, which varies from polymer to polymer, and because of the diverse nature of the compounds used as flame retardants" (Kasem & Rouette, 1973, p.36). Essentially, the application of a fire retarding agent to a textile changes the normal course and speed of the combustion reaction favoring pathways which depress combustion parameters. Materials treated with a fire retardant agent are, thereby, more difficult to ignite, burn more slowly, have lower heat emission or are more easily extinguished than the untreated material. Combustion of fire retardant treated cellulosic textiles commonly leads to the production of more char and less flammable gas (Kasem & Rouette, 1973; Lindsay, 1966). All known fire retardants lower the decomposition temperature of cellulose (Reeves & Guthrie, 1966), consequently, reactions favoring dehydration and charring will predominate.

Several theories have been advanced for the general mechanisms by which fire retarding agents may function. These are reviewed in a number of publications (Chamberlain, 1978; Drake & Reeves, 1971; Gottlieb, 1956; Little, 1951; Lyons, 1970). It is generally accepted that there are two major categories into which the theories of function can be divided; categorization is by physical mechanisms or by chemical change. The physical functioning theories suggest that in the event of combustion the fire retarding agent may act in one of the following ways:

1. decompose to produce a glass-like coating over the textile fibers thereby trapping volatile tars and excluding the oxygen necessary for the burning process.
2. decompose with the evolution of non-combustible gases thereby diluting the flammable gases produced by the burning cellulose.
3. absorb or dissipate heat by an endothermic change in its compostition, such as fusion or sublimation, the endotherm preventing propagation of combustion (Drake & Reeves, 1971).

According to Kasem and Rouette (1973) these theories are applicable to a limited number of fire retarding agents, and the chemical theory of catalytic dehydration is more generally applicable.

The catalytic dehydration theory assumes that a fire retardant is a Lewis acid or its precursor (Kasem & Rouette, 1973; Reeves & Guthrie, 1966). Lewis acids have been defined as electron-pair acceptors (Solomons,1976). This means that they are capable of forming a chemical bond with electron pair donors such as the hydroxyl groups (OH) of the cellulose molecule. A hydroxyl group is drawn away from the cellulose chain by the hydrogen ion ($H^{\cdot}$) of the acid and a molecule of water is eliminated. As successive hydroxyl groups are removed from the cellulose molecules a skeletal chain high in carbon content remains. The dehydration can proceed either by carbonium ion catalysis according to following scheme:

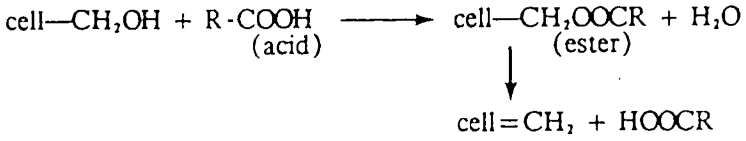


$$-\underset{\underset{H}{|}}{\overset{|}{C}}-\underset{|}{\overset{|}{C}}-OH \xrightarrow{H^{\cdot}} -\underset{\underset{H}{|}}{\overset{|}{C}}-\underset{|}{\overset{|}{C}}-OH_2^{\cdot} \longrightarrow -\underset{\underset{H}{|}}{\overset{|}{C}}-\underset{|}{\overset{|}{C}}^{\cdot} + H_2O \quad (1)$$

$$\downarrow$$

$$\underset{|}{\overset{|}{C}}=\underset{|}{\overset{|}{C}} + H^{\cdot}$$

or by esterification and subsequent decomposition of the ester as follows



$$\text{cell}-CH_2OH + \underset{\text{(acid)}}{R\text{-}COOH} \longrightarrow \underset{\text{(ester)}}{\text{cell}-CH_2OOCR} + H_2O \quad (2)$$

$$\downarrow$$

$$\text{cell}=CH_2 + HOOCR$$

(Lewin and Basch, 1978)

No single theory adequately explains the function of all fire retardant agents, nor can it be assumed that there is only one mechanism involved. Fire retardation essentially interrupts the burning process at one of the critical junctures of combustion (Pitts, 1973). These junctures are fuel production, ignition, flaming combustion and glowing combustion. According to Chamberlain (1978) "the dominant if not the only mechanism of fire retardance for cellulose...is alteration of fuel production" (p.122). Catalytic dehydration of cellulose by a Lewis acid through a carbonium ion or esterification mechanism directs the path of cellulose decomposition towards the production of carbon char and water and away from the production of flammable tars and gases. According to Barker and Hendrix (1978) "by catalysing the degradation so that it would occur at a temperature below the minimum required ...for levoglucosan formation, tar formation and flaming should be reduced" (p.34).

In conclusion, it should be noted that fire retardant agents are "useful during the ignition, flame spread and growth phases of a fire where the incident heat flux on the bulk of the fuel load is low. Fire retardant agents will have no useful function in the later stages of a fire where incident heat fluxes are very high" (Chamberlain, 1978, p.114).

2.7 Fire retardant chemicals for cellulosic textiles

As discussed previously, cellulosic fibers are naturally highly flammable, but "fortunately cellulose has a chemical composition which makes it particularly amenable to treatments which can render it both flaming and glowing resistant" (Barker & Hendrix, 1979, p.1). Treatments can be applied to the cellulose substrate in either an additive or reactive manner; that is, the fire retardant chemical may be deposited on the fiber structure as a separate chemical substance or it may undergo a chemical reaction with the cellulose substrate and become a part of its chemical make-up.

Fire retardant chemical agents are generally classified by their durability (Drake & Reeves, 1971; Lewin & Sello, 1975; Marsh, 1957). There are non-durable finishes which are water soluble additive compounds, usually inorganic salts; semi-durable finishes which are precipitated as insoluble salts on to the substrate; and durable finishes which react with the fibers at the molecular level or are bound to them by crosslinking agents or resins.

Non-durable treatments are applied from a water solution by dipping, spraying or brushing. Because of their water soluble nature they are removed by washing, or leaching by moisture in the air. They are also very vulnerable to leaching by moisture in the environment. This latter problem is well illustrated by Clark and Dillon in *Preliminary Investigation of a Large Modern Wallhanging*, 1981 (Note 3).

Semi-durable and durable treatments require two or more steps in order to be durably fixed to the cellulose substrate. A double bath process is commonly employed. In between the two bath dips is a squeezing step which aids penetration of the treatment and removes excess moisture. Heat, at a temperature specific for a particular treatment, is frequently employed as a curing process; this step is considered to be crucial in successful application of a durable finish (Yeager & Chapin, 1972). "For reactive-type flame retardants the exact cure time and temperature are critical and must be reproducible within narrow limits" (Miles & Delasanta, 1968). Other finishing treatments may require a step such as an ammonia gas cure; most processes require a final wash. A one step process in which a durable fire retardant was applied from an organic solvent was developed. It could be applied in the field by painting or spraying (Drake & Reeves, 1975; Miles & Delasanta, 1968). Unfortunately it was based on the use of tris(2,3-dibromopropyl) phosphate which has since been banned.

According to information supplied by the Edmonton Fire Marshall's office the fiber art works that are required to be fire resistant are those that constitute more than 10% of the wall space they occupy in a public building. This figure is derived from flame spread regulations in the Alberta Building Code, Section 10.4.3 wherein it is stated that 90% of the wall surface area must have a designated flame spread rating. The unique nature of fiber art works, their frequently monumental scale, and the possibility of in-situ construction all preclude the use of finishing processes that require complex equipment and/or specific, controlled conditions for successful application should a fire retardant finish be necessary. Whilst precipitation of insoluble salts may be possible this type of treatment adds greatly to fabric weight and has highly degradative effects on the fibers (Lindsay, 1966). It would appear, therefore, that at present non-durable fire retardants, which can be applied easily and successfully in the field, are likely to be the most useful to the fiber artist.

Whilst many inorganic compounds can be expected to prevent flame propagation if present in large enough amounts, they are not generally regarded as efficient agents due to the excessive add-on required which has detrimental effects on the character of the textile (Marsh, 1957). Also, many effective flame retardants do not prevent afterglow. "In practice, only a few very efficient agents, or mixtures of such, are used which are capable of imparting a high degree of resistance to both flaming and afterglowing" (Lewin & Sello, 1975, p.21). Six elements have been identified as being the most important in fire suppression. These are phosphorus, antimony, chlorine, bromine, boron and nitrogen (Lyons, 1970). Phosphorus compounds are the most effective glow retardant agents. The chemistry of inorganic fire retardants for cellulosic fibers is explored fully in Barker and Hendrix, (1979); Lewin and Sello, (1975); and Pitts, (1973). It has been shown that chemical reactions modifying the cellulose molecule at the C-6 position suppresses the formation of levoglucosan and thereby leads to reduced flammability (Barker & Hendrix, 1979).

Those agents which appear to be most viable for use on fiber art and which are available commercially are to be found in the non-durable group. Of significance are the borates, the phosphates and the sulfates, the compounds which have the longest history of use. In addition, the halogens, chlorine and bromine in the form of secondary additives have proven useful (Lyons, 1970). Ammonium compounds are the ones most frequently used, their main advantage being ready decomposition on heating to give an inorganic acid which acts as the fire retardant. There is also the liberation of a considerable amount of ammonia gas which is thought to act as a diluent to the combustible gasses (Pitts, 1978). Other work, however, indicates that the role of ammonia may be negligible in the overall resistance to fire (Kishore and Mohandas, 1983).

The most frequently encountered mixture is that of boric acid and its sodium salt, borax, used in varying proportions. According to Pitts (1973), boric acid is considered to be one of the best afterglow suppressants, but there appears to be variations in effectiveness depending upon the ratio of the borax/boric acid formula and the percentage add-on achieved (Lyons, 1970). The addition of ammonium phosphate to the borax/boric acid mixture has been advised as an afterglow suppressant (Lewin & Sello, 1975; McKinnon, 1976). It is generally accepted that the borates operate by a physical mechanism of melting at relatively low temperatures and forming a glass-like coating over the decomposing textile fibers, preventing combustion by excluding oxygen. There is now evidence that a chemical mechanism is also operative. It is suggested that the borates can catalyse an esterification reaction leading to a change in thermal degradation products (Pitts, 1973; Shafizadeh, 1968).

Phosphates are perhaps the most versatile of the usable agents. Whilst their use is historical, many present day durable treatments of significance are based on phosphorus compounds in a synergistic relationship with nitrogen. Ammonium phosphate and diammonium phosphate are the most widely used non-durable fire retardants; they decompose on heating to give ammonia and phosphoric acid (Lewin &Sello, 1975). Phosphate retardants function by altering fuel production during pyrolysis. They appear to operate through several mechanisms, although the relatively low amount of ammonia produced apparently rules out a gas dilution function (Pitts, 1973). Liberated phosphoric acid directs the decomposition reaction towards the production of char and away from the production of levoglucosan. There is evidence supporting both the carbonium ion and ester formation reactions as a means of achieving this result (Pitts, 1973). As temperatures increase it is suggested that a polyacid is produced which is capable of forming a viscous, glassy melt that forms a film over the char. The film so formed "would retard the diffusion of oxygen into the reactive layer of carbon" (Pitts, 1973, p.145) resulting in the elimination of afterglow. It should be noted that this theory has been discounted by others who support a theory of inhibitors interfering with production of oxides of carbon (Chamberlain, 1978).

Sulfates have the longest history of use in the search for fire retardant finishes. The first known use of sulfates was in Roman times; and it was mentioned in a scientific context in 1638 in the first published text on flame resistant treatments for textiles (Pitts, 1973). The compounds of interest in this group are ammonium sulfate and ammonium sulfamate. Ammonium sulfate operates in a similar manner to ammonium phosphate , that is, through the liberation of ammonia and an acid. Ammonium sulfamate was introduced commercially as a fire retardant for cotton textiles over forty years ago, and is still found in commercial formulations. Its action is thought to take place in the gas phase through the endothermic evolution of ammonia and sulfur trioxide gas. Neither compound imparts glow resistance. Sulfur based chemicals have been found to be equally effective on cotton and rayon, whilst phosphorus based compounds are less effective on rayon than on cotton (Lewin & Basch, 1975).

According to Lyons, (1970) "there appear to be two or three main classes of flame retardant treatments for cellulose fibers for which there is a significant commercial demand. These [include] the temporary water soluble types...where contact with water is deemed unlikely,...[This] class of uses is well served by the soluble ammonium phosphates and the borates" (p.229).

2.8 Characteristics of a suitable finish for cellulosic fiber art

When considering the issue of fire retardant finishes for fiber art there are two opposing needs or requirements. On the one hand the law requires that a finish be applied in specific situations and, on the other hand, the textile conservator is concerned for the preservation of the textile through the elimination of foreign substances in contact with that textile. In the hanging of an art textile in a public building the artist has no choice: the textile art work must conform to the regulations either through inherent characteristics or, failing that, through the application of a finish. If a finish is found to be necessary it becomes imperative, from a conservation point of view, that the finish selected be as non-damaging to the art work as possible. The following points should be considered:

2.8.1 Ease of application

This is a primary consideration. The "one-of-a-kind" nature of a fiber art work, three-dimensional structure and in-situ construction all preclude the use of complex mechanical equipment in the application of a finishing treatment. Spraying, dipping or brushing methods of application can be used, but mechanical squeezing or high temperature drying would be damaging to the textile fibers.

2.8.2 Minimum add-on

The chemical formulation used should impart flame and glow resistance with a low % add-on of the finishing treatment. Add-on of chemicals increases the weight of a fiber piece. Should the art work be large and bulky too much added weight would cause considerable strain on supporting points. High add-on levels may also create negative effects on hand and drape such as making fibers stiff and sticky. These effects will be particularly noticeable in a lightweight structure. A sticky surface will hold on to damaging dirt in the environment.

2.8.3 Dimensional change

If the fiber art work is in its finished form the application of a finishing treatment should not alter the dimensions in any way, otherwise the artistic concept is altered. As the finishes suitable for fiber art application are generally applied from a water solution, pre-shrinking of yarn and fabric may be necessary (Platus, 1977).

2.8.4 Compatibility with dyes

It is essential that the fire retardant finish not alter the colour of dyes because of the importance of colour in the aesthetics of art. It is also necessary that the fire retardant compound be compatible with any other finishing treatments used. Suzuki (Note 4) advised of problems of associating a fire retardant finish with Scotchgard®. The finishing treatment was said to have been rendered ineffective by the Scotchgard®.

2.8.5 Freedom from degradative action

The pH level of a fire treatment is of major importance from a conservation standpoint. Cellulosics are particularly vulnerable to degradation by acids, therefore the finish should be either neutral or alkaline (Lyons, 1970).

2.8.6 Permanency to display conditions

This is necessary from a regulatory and safety point of view. The fiber artist should be aware of the conditions, that is the temperature, relative humidity and lighting levels under which the fiber art work will be displayed. Environmental conditions should be adjusted wherever possible to conform to prescribed optimum levels for the long term benefit of the textile fibers (Lafontaine, 1979). The finish should have maximum durability under display conditions, and in particular it should not be vulnerable to leaching under prevailing humidity levels.

2.8.7 Durability

It is highly desirable that a finish have some durability to cleaning processes.

2.9 Application and effects of fire retardant treatments

The application of water soluble finishes to cellulosic fiber art work can be accomplished by immersion of the textile in the solution or by spraying or brushing the solution on to the textile. "The particular method used and the proportion of the water used in the solution is not important as long as uniform treatment and required add-on are obtained" (McKinnon, 1976, p.3-10). There must be contact between the molecules of the finishing agent and the textile fibers, thus fabric penetration is an important requirement, as is uniform penetration (Valko,1971). In order to get a uniform spread the chemicals must be applied in a diluted form to fibers that are absorbant (Lindsay, 1966; Valko, 1971).

"Pure water is incapable of spontaneously and completely wetting capillaries of organic polymers, with the possible exception of cellulose that has been conditioned at sufficiently high relative humidity" (Valko,1971, p.93). In the case of fiber art work it is unlikely that conditioning would be possible, in which case the addition of a wetting agent to the finishing treatment will act to reduce surface tension of the water and allow the solution to penetrate the fabric sufficiently.

If the finishing treatment is applied through immersion of the textile in a bath, some suitable method must be found for removing excess moisture. The usual methods of squeezing between rollers or centrifugal force are not practical or safe for fiber art. Hanging to drip dry would not only be detrimental to the art work but it would also cause migration of the finishing chemicals to give a higher concentration at the lower portion of the textile. Blotting and horizontal drying should be employed if immersion application is used (McKinnon, 1976).

Valko (1971) states that "if a liquid in the form of droplets (aerosol) is sprayed onto a fabric in sufficient amount and wets the fiber surface, capillary migration tends to distribute the liquid uniformly through the fabric" (p.107). It is suggested that "this is a practical method for impregnating the fabric with a treating solution without immersing it into an excess volume of bath and without the need of squeezing out excess liquid" (Valko, 1971, p.107).

Finishing treatments should be dried carefully. Fast drying may result in the crystallization of chemicals on the surface of the textile. In the case of ammonium salts, too high a drying temperature might result in the loss of ammonia through the decompostion of the compound by heat (Lewin & Sello, 1975; Lyons, 1970). If a finish of diammonium phosphate is exposed to too high a heat in drying the loss of ammonia will lead to increased acidity of the fabric which will cause it to tender (Lyons, 1970). In the application of the borax/boric acid mixture it is suggested that drying at a temperature below 100 C helps retention of fiber tensile strength (Lyons, 1970). Ammonium sulfamate has been found to leave fabrics with a desirable hand (Lewin & Sello, 1975; Marsh, 1957), although it has also established a reputation for tendering the fabric (Marsh, 1957; Pitts, 1973). It is suggested that strength properties of the textile are not affected if drying of a sulfamate finishing treatment is kept below 70 C and the treated article is not exposed to strong light for an extended period of time (Lewin & Sello, 1975). It is also indicated that sulfamate requires the use of buffering agents, such as trisodium phosphate, to prevent the yellowing effects of light (Lewin and Sello, 1975).

Fire retardant finishing agents have a history of causing a reduction in tensile strength, and changes in colour, hand and drape of fabrics (Drake & Reeves, 1971; Kasem & Rouette, 1973; Lindsay, 1966; Lyons, 1970). Much has been done to modify these negative effects. Nevertheless, they have not been totally eliminated. Care during the curing process is critical to maintaining fabric tensile strength when semi-durable and durable treatments are applied (Drake & Reeves, 1971). The fiber artist may encounter these types of finishes when using commercially produced fabrics in an art work. Commercial plants have the equipment necessary to produce fire retardant treated fabrics under optimum conditions. It is to be expected, therefore, that these fabrics will exhibit minimal change in properties as a result of their fire retardant treatment.

According to McKinnon (1976) most treatments in use today do not result in a reduction of tensile strength under normal use, nor will colour and hand be unduly affected by most treatments. This comment, however, is at variation with other researchers such as Vail, Daigle and Frank (1982) who record some adverse effects depending upon treatment or method of application. Nor does it take into account lack of control associated with the application of non-proprietary mixtures or commercials reagents by unskilled personnel. Exposure to high temperature and light reduces fiber strength, with sunlight being particularly damaging (Drake & Reeves, 1971; Lyons, 1970; Marsh, 1957). Of the inorganic soluble compounds considered suitable for fiber art, borax and ammonium phosphate have been found to have the least destructive action (Marsh, 1957). It is suggested that a boro-phosphate combination of fire retarding agents will afford good fire resistance properties without the disadvantage of acidity (Marsh, 1957). Lindsay (1966) advises the addition of a small quantity of ammonia to a formulation to neutralize any residual acids.

2.10 Fire codes

Historically, regulatory measures related to fire safety and the combustibility of textiles in public buildings have become more restrictive with time. There are localized differences in codes and in application and enforcement of the codes, because there is no single legislated code for the whole of Canada. The National Fire Code of Canada (1980) is a "model set of technical requirements designed to provide an acceptable level of fire protection and fire prevention within a community...written in a form suitable for adoption by appropriate legislative authorities in Canada" (p.vii). It is being used as the foundation of the new Alberta Fire Code under consideration at this time. The Alberta Fire Code is expected to be passed into law by early 1984.

The requirements for interior textile finishings will almost certainly be the same as those set down in the National Code. The section relevant to fiber art is Section 2.3 Interior Finishing, Furnishing and Decorative Materials, Subsection 2.3.2. which is quoted in full.

SUBSECTION 2.3.2. FLAME SPREAD

2.3.2.1.(1) No person shall install drapes, curtains and other decorative materials including textiles and films used in buildings that do not meet the requirements for a high degree of flame resistance as described in Note 4 of Method 27.1 of CAN2-4.2-M77, "Textile Test Methods", when such drapes, curtains and other decorative materials are used in

(a) any Group A or Group B [1] occupancy that contains a room or an area that has an occupant load of more than 100 persons,

(b) any lobby or exit, or

(c) any open floor areas in any Group D, E and F occupancy exceeding 500 sq.m., except when the floor area is divided into fire compartments not exceeding 500 sq.m in area and separated from the remainder of the floor area by fire separations having at least a 1 h fire-resistance rating.

2.3.2.2.(1) Flameproofing treatments shall be renewed as often as is necessary to ensure that the material referred to in Article 2.3.2.1. will pass the match flame test in NFPA 701-1977, "Standard Methods of Fire Tests for Flame Resistant Textiles and Films".

An additional requirement is proposed for the Alberta Fire Code.

"A written record of any tests and/or flameproofing treatments carried out shall be maintained by the owner for a minimum of two years".

The Alberta Fire Prevention Act (Assented to May 4, 1982) Chapter F-10.1 delineates enforcement procedures. Under section 11.2.d a person authorized to make an inspection may "perform or cause to be performed any tests he considers necessary on the building...or anything in it, and may remove anything for the purposes of the inspection" (p.5). Under section 13 if "the person making the investigation or inspection is of the opinion that a condition of emergency exists due to a fire hazard...he may not withstanding anything in this Act, forthwith take any steps he considers appropriate to remove or lessen the hazard or risk" (p.7).

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[1] for classification descriptions see Alberta Building Code 1981, Section 3.1

2.11 Toxicity

This aspect of fire retardancy is complex and cannot be explored in any depth in this review. It is an important factor for the fiber artist to consider, however, particularly in light of the problem with tris(2,3-dibromopropyl) phosphate. "The 'Tris' experience suggests that exposure and health hazard may be critically dependent on specific properties of the textile substrate, on the mode of incorporation of the compound in the substrate, on the interactions (including chemical reactions) of the compounds with the substrate and on the conditions of processing" (Tesoro, 1979, p.243). There are two aspects of toxicity to be considered; they are the hazard potential of chemicals in the application process and the hazards associated with the decomposition products during combustion of the treated textile.

The chemicals which are considered suitable for fiber art applications have either very low levels of toxicity or no toxicity rating at all (Windholz, 1976). **Ammonium sulfate** and the **ammonium phosphates** do not have toxicity problems. **Ammonium sulfamate** has a very low order of toxicity; ingestion may cause gastro-intestinal disturbances. Ingestion or absorption of very high amounts of **boric acid** can cause death (5-20g for adults, <5g for children). Lesser amounts or chronic use may cause gastric disturbances, dry skin and eruptions. Ingestion of 5-10g **sodium borate (borax)** by young children can cause severe vomiting, diarrhea, shock or death.

According to McKinnon (1976) "except for some of the more complex compounds used in durable treatments, few of the chemicals in common use have any irritating or toxic properties, and there are rarely any problems encountered in the handling of most chemicals or treated fabrics" (p.3-10). Should the fiber artist find it necessary to apply fire retardant treatments personally, sensible handling and storage make them safe to use. It is always wise to protect the hands with gloves and the respiratory system with a mask.

Fire retardant treatments are designed to alter the process of thermal decomposition of the cellulose substrate. Whilst this change leads to a reduction in the flammable components of the combustion process, it cannot be assumed that there will be a reduction in toxic byproducts (Uehara & Yanai, 1973). According to Lewin and Sello (1975) "little is known about the influence of flame retardants on the production of toxic gases in the combustion of cellulosics" (p.11). For example, evidence has been found for both a reduction in carbon monoxide levels (Lewin & Sello, 1975), and for an increase in carbon monoxide levels (Hilado

& Brauer, 1979). Also the degree of hazard may not necessarily be explained by the concentrations of individual chemical components.

Data on individual compounds cannot be used to assess toxicity hazards since a mixture of toxic gases is usually produced in fires and synergistic effects are to be expected. For example, the presence of carbon dioxide increases respiration thereby accelerating the uptake of such substances as carbon monoxide which has a high affinity for hemoglobin (Lewin & Sello, 1975). Research has shown that during combustion a fabric treated with a fire retardant agent tends to produce greater amounts of smoke and gas than an untreated fabric (Uehara & Yanai, 1973). Because both smoke and toxic gases occur together it is difficult to separate the effects of the two; both are easily able to cause asphyxiation.

Smoke is a double hazard in that it also reduces visibility. Where burning textiles are concerned, smoke is a greater hazard than toxic gases. The results of smoke emission tests show that the amount of smoke emitted from the burning of fire retardant treated cotton is nearly twenty five times the amount emitted from the burning of untreated cotton (Benisek, 1975). These figures are based upon measurements of decreased visibility; the type of fire retardant treatment was not mentioned. Diammonium phosphate has been shown to affect smoke levels in a complex manner depending upon the concentration of the compound and the temperature to which it is exposed (Uehara & Yanai, 1973).

2.12 How fire retardancy issues affect the fiber artist

In accepting a commission to produce a fiber piece to be placed in a public building the artist should accept responsibility, not only for the aesthetics, but also for the care and preservation of the work. To do otherwise would display a lack of professional committment unless the work is specifically planned for obsolescence, in which case there should be an understanding between the artist and the client from the very beginning (Constantine & Larsen, 1981).

Whilst ultimate legal responsibility for the fire retardancy of a fiber piece in a public building rests with the owner of the building, it is in the artist's best interests to accept responsibility for seeing that the work conforms to standards set by local fire codes. That this should be done with the best interests of the textile in mind is the rationale for this study. Lack of information on codes and finishes appears to be a major problem (Platus, 1977).

Personal communication with fiber artists has illustrated that the issue of fire retardancy is usually acknowledged but not always dealt with in an informed manner. In the handbook *Commissioning Textile Artworks* (Note 5), Thomas raises the issue and places the onus on the artist. However, the references only guide the reader to the regulations. A United States artist, Ove (1978) expressly states that she was "motivated to experience some of the realistic conditions involved in designing and executing a large textile hanging for a specific public building" (p.1), but nowhere was the issue of fire retardancy mentioned.

When it becomes necessary to apply a fire retardant finish to an art work the artist has three viable alternatives. These are self application of a non-proprietary mixture, self application of a commercial reagent and commercial application. Three artists who have worked on large scale pieces, Platus (1977), Watson,(Note 6) and Suzuki, (Note 4) have found it preferable to contract the work out to commercial concerns. It should be noted, however, that these artists work in centres large enough that knowledgeable, commercial treatment personnel are available.

McKinnon (1976) warns against the use of commercial concerns of unknown reliability because it has been found that "there are some concerns in this field which have not treated fabrics properly or have used relatively ineffective chemicals" (p.3-10). Even where commercial firms are knowledgeable of fibers and chemicals they are frequently unfamiliar with fiber art (Platus, 1977). Similarly, fiber artists who are trained in the aesthetics and technology of their medium are rarely knowledgeable of the chemicals used as finishing treatments. McKinnon (1976) concludes that "with few exceptions, flame retardant treatments can be entrusted safely only to skilled persons" and as a final admonition states "it is ...a field which is especially vulnerable to entry by unskilled entrepreneurs and charlatans. Let the buyer beware!" (p.5-70).

In a 1978 study of the fiber arts market in the southern United States Davis(1979) indicated that the problems of meeting "flameproofing" standards constituted a significant barrier to the commissioning of fiber art by commercial architects.[2] She recommended that fiber artists become more familiar with "textile" properties because fiber content of the art work is a major factor in meeting flameproofing policies. She further recommended increased knowledge of flameproofing finishes and local and federal flameproofing regulations.

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[2] commercial architects are defined as those whose work mainly constitutes designing public buildings

It was suggested that ease of meeting flameproofing policies should be investigated by fiber artists as a means of increasing their market potential.

3. PROCEDURE

3.1 Materials

Each fiber art work is unique. In developing an artistic concept for a specific commission the artist will combine dyes, fibers, yarns and/or fabrics in a manner unique to that individual construction. Since scientific study requires a set of conditions that are controllable and reproducible, a commercially produced test fabric was used as the cellulosic substrate for this research study. This choice of fabric allows for a direct comparison of test results between the control samples and the samples treated with different finishes.

Selection of the fire retardant finishes used in the study was based upon the availability of commercial reagents and upon the chemical compounds which a review of the literature showed to be the most likely to be used on fiber art.

3.1.1 Test fabric

The fabric used for treatment and testing is a plain weave, unbleached, desized 100% cotton print cloth, having a weight of 120g/m^2 and a yarn count of 35x32 per cm (warp x weft). It is designated style 400U by the manufacturer, Testfabrics Inc., Middlesex, NJ.

3.1.2 Fire retardant finishes

Two commercial reagents and one non-proprietary mixture were used to give three different sample treatments which were compared with an untreated control sample. The treatments were as follows:

1. A mixture of borax, boric acid and diammonium phosphate in proportions of 7:3:5 mixed with 110 parts water. This is formula #2 in *Fire Protection Handbook* (McKinnon, 1976, p.3-10).
2. X-12 supplied courtesy of Spartan Adhesives and Coating Co., Crystal Lake, Illinois. Ownership and manufacturing rights of this product were recently acquired from E.I. du Pont de Nemours and Company. Due to the proprietary nature of this reagent no formula could be obtained. Information from accompanying technical literature suggests that the major component is ammonium sulfamate.

3. FlameGard DSH produced by Jersey State Chemical Company/Sybron Corporation, Haledon, N.J., and supplied courtesy of Tanatex/Sybron Canada Ltd., Downsview, Ontario. The company would only indicate that these proprietary compounds are a blend of inorganic ammonium salts.

For chemical analyses of X-12 and FlameGard DSH see Appendix I. The analyses were done courtesy of the National Research Council's Edmonton, Alberta laboratory.

Control samples were immersed in distilled water and the same wetting agent, JWet FR, used with above treatments. It was supplied courtesy of Tanatex/Sybron Canada Ltd. The only difference between the control and treated fabrics was in the fire retardant chemicals applied.

3.2 Methods

3.2.1 Sample preparation

The ultimate objective of the physical testing of textiles is to predict behavior in use. In order to obtain reliable results samples taken from the test fabric must be representative of the whole. When accelerated aging is carried out the end product of the fabric aging process should be similar to that which results from natural aging even though the route may be different.

3.2.1.1 Cutting procedures:

Prior to treatment four pieces of test fabric 150cm long and 70cm wide were cut from the bolt and marked with control and treatment designations. In addition, measurements for dimensional change in wetting, as specified in Can 25.1 (CGSB 1977) were marked on the fabric in grey silk thread. No piece was cut within 10cm of the selvedge or within 5cm of either end of the bolt. After treatment, samples for each series of tests were cut so that each sample in a test had a different set of warp and weft yarns. No sample was cut within 5cm of any edge of the treated fabric with the exception of samples required for establishing fire retardant effectiveness.

3.2.1.2 Dyeing procedure:

Coloured samples were prepared by dyeing swatches of test fabric with Procion® fiber reactive dyes in six colours. They were red, yellow, blue, orange, green, and purple. The method used for dyeing was the long method for Procion® M-Series dyes shown in Appendix II.

3.2.1.3 Accelerated aging procedure:

Accelerated aging provided controlled, reproducible conditions of degradation required for the study. The conditions used in the accelerated aging process did not necessarily simulate conditions of use for fiber art, but provided uniformly degraded material suitable for evaluating and comparing the effects of the different fire retardant treatments. The accelerated aging process involved exposure of test specimens to a temperature of 60-65 C, and a relative humidity of 20-35%. Continuous light was provided by a carbon arc in an Atlas Weatherometer model 18-WR. The aging technique was designed to impart a loss of tensile strength of about 50% to the control fabric and required an exposure time of 495 hours.

3.2.2 Treatment preparation and application

The aim of the fire retardant treatment was to enable the test fabric to pass the vertical flame test as prescribed by the National Fire Code of Canada (1980). Preliminary experimentation with X-12 and FlameGard DSH indicated the appropriate concentration of solution required to provide sufficient add-on of chemicals for treated fabrics to pass the test in one application. The concentration of the non-proprietary reagent, borax:boric acid:diammonium phosphate, as specified in *Fire Protection Handbook* (McKinnon, 1976), was adhered to and sufficient add-on achieved by repeated applications.

It should be noted here that the level of add-on required for the fabric to pass the vertical flame test is specific to the type and weight of test fabric used. A heavier weight or closer weave fabric would require a lower add-on than a lighter weight or looser weave fabric.

Treatment preparation and application was as follows:

Control: warm distilled water (35 C) with the addition of 1% JWet FR (wetting agent).

- solution pH - 7.6
- application procedure - immersed/dried

Borax/boric acid/diammonium phosphate: 7:3:5 parts in 110 parts by weight distilled water with the addition 1.5% JWet FR (wetting agent), (McKinnon, 1976, p.3-10). Distilled water was heated to 65 C. The boric acid mixed with a small amount of water was added along with the remaining chemicals and wetting agent to the hot water and stirred until dissolved leaving a clear solution. It was allowed to cool before application.

- solution concentration - 13.6% (w/w)
- solution pH - 8.4
- application procedure - immersed/dried/immersed/dried

Testing at this juncture indicated a 13% add-on but the fabric was unable to pass the vertical flame test. A further immersion and dry followed which enabled it to pass the flame test.

- add-on - 15%

X-12: prepared using distilled water heated to 80 C in accordance with the manufacturer's specifications. An addition of 1% JWet FR (wetting agent) was used for experimental control. The solution was cooled prior to application.

- solution concentration - 18% (w/w)
- solution pH - 6.7
- application procedure - immersed/dried
- add-on - 15.2%

FlameGard DSH: prepared using distilled water at 35 C and 1% JWet FR (wetting agent) according to manufacturer's specifications.

- solution concentration - 20% (v/v)
- solution pH - 8.2
- application procedure - immersed/dried
- add-on - 10.7%

The temperature of the treatment solutions upon application was about 27 C. The equipment used for the application of the finishing treatments consisted of a plastic trough, sold for the purpose of wetting out wallpaper, and a piece of ABS two and a half inch diameter sewer pipe cut to the length of the trough. Interior measurements of the trough were 75cm long, 12cm wide and 9cm deep. Inside the trough was a plastic rod 3cm from the base and 3cm from one side which formed a channel for pulling the wetted material through. The prepared treatment solution was placed in the trough and the leading edge of the fabric was rested on the rod. The ABS pipe, which acted as a roller, was then placed on the top of the fabric and the fabric was gently pulled through onto a flat table top protected with white cotton terry cloth. The treated fabric was allowed to dry aided by a cool air fan. When completely dry it was rolled onto a Mylar protected cardboard tube and covered with acid free tissue.

As preliminary work had indicated the borax:boric acid:diammonium phosphate (7:3:5) treatment had a tendency to crystallize out onto the surface of the fabric at 65% RH it was decided to store all the treated fabrics under ambient conditions until required for testing.

3.3 Test Methods

3.3.1 Mass of Fabrics

The purpose of test Method 5.A-1977 (CGSB, 1977) in this study was to determine the amount of finish deposited onto the test fabric during treatment with the fire retardant solutions. The treated fabric was conditioned at 65±2% RH and 21±2 C. Five die cut specimens were taken, no two specimens containing the same warp or weft yarns. Specimens were weighed on a Sartorius balance. The difference between the weight of the control fabric and the weight of the treated fabric expressed as a % of the control fabric, indicated the level of add-on, or the increase in weight due to the presence of the finish.

3.3.2 Flame Resistance - Vertical Flame Test

Samples of treated fabric were tested following the procedure prescribed in Method 27.1-1977 (CGSB, 1977) using the equipment shown in Appendix III. Flame resistance is evaluated on the basis of flashing of flame over the surface, afterflame, afterglow which can occur once the flame is extinguished and length of char. In general a high degree of flame resistance is indicated by absence of flashing, an average afterflame of less than 2 sec, afterglow not extending beyond the area originally charred by the flame and an average char length not exceeding 90mm. It is recognized that the average acceptable char length may vary with the mass and type of fabric being tested.

3.3.3 Dimensional Change in Wetting

The fabric was conditioned at 65±2% RH and 21± 2 C prior to treatment. Measurements were marked as prescribed in Method 25.1-1977 (CGSB, 1977) using grey silk thread. Following treatment the fabric was reconditioned and remeasured without pressing. Six measurements were made in the warp direction and six in the weft direction. Dimensional change was calculated as the percent difference between the average of the six measurements taken before treatment and the average of the six measurements taken after treatment.

3.3.4 Fabric Tensile Strength

Test specimens were conditioned at 65±2%RH and 21±2 C prior to testing. Tensile strength of the fabric was measured using the prescribed Method 9.1-1977 (CGSB, 1977) but with a reduced specimen size and samples cut in the warp direction only. The size of the cut specimens was 150x20mm and they were ravelled to 50 warp ends. Tensile strength was measured on an Instron(CRE) machine using a 75mm gauge length, and a load range of 0-20kg. The cross head speed was varied from 1 - 3cm/min to provide an average time to break of 20±3 secs.

3.3.5 Stiffness of Fabrics

Test specimens were conditioned at 65±2% RH and 21±2 C prior to testing. Fabric stiffness, also known as flexural rigidity, was measured as prescribed in ASTM D 1388-64, Option A - Cantilever test (ASTM, 1983). Flexural rigidity is calculated as follows:

$$G = c^3 w \qquad (1)$$

where G = flexural rigidity (mg/cm), c = bending length (cm), and w = mass(g/m^2)
The overall flexural rigidity of a fabric is calculated by the formula:

$$G_0 = (G_w G_f)^{1/2} \qquad (2)$$

where G_0 = overall flexural rigidity, G_w = warp flexural rigidity, and G_f = weft flexural rigidity.

Bending length is "a measure of the interaction between fabric weight and fabric stiffness as shown by the way in which a fabric bends under its own weight. It reflects the stiffness of a fabric when bent in one plane under the force of gravity, and is one of the components of drape." (ASTM, 1983, p.295). The test was repeated with the same samples under ambient conditions of 21% RH and 22 C.

3.3.6 Fabric pH

The pH of the fabric was recorded using an aqueous extract technique. Aqueous extracts were prepared according to Method T 509 os-77 (TAPPI,1977) Cold Extraction Method, but with reported results being an average of seven determinations for unaged cotton and two for aged cotton. The readings were recorded with a Fisher Accumet Model 230 pH/ion meter using a E-5D combination electrode.

3.3.7 Fabric Colour Change

The colour of undyed unaged specimens, dyed specimens, and undyed aged specimens was measured and colour differences between the water treated control fabric and treated fabrics were calculated in CIELAB units. A Hunterlab Model D25M/L tristimulus colorimeter with microprocessor was used for these measurements and calculations. The *L* value is a measure of lightness and darkness; *a* is a measure of the degree of redness or greeness (*+a* is a red value and *-a* is a green value); *b* is a measure of the degree of yellowness or blueness (*+b* is a yellow value and *-b* is a blue value). The colour difference (Δ E) is calculated by the following CIE 76 $L^*a^*b^*$ colour difference formula:

$$\Delta E \text{ (CIELAB units)} = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

(AATCC, 1982/3)

4. RESULTS AND DISCUSSION

This chapter will commence with a description of the statistical analyses performed on the numerical data resulting from the tests performed. The results will then be discussed in relationship to the findings of other researchers wherever possible, keeping in mind the conservation focus of the study and the importance of the findings to the fiber artist. Significant changes occuring in the variables of dimension, tensile strength, stiffness, pH and colour as a result of treatment and accelerated aging procedures will be identified.

In general, it was found that each test performed to determine changes of importance to the aesthetics and preservation of fiber art indicated that treatment with a fire retardant finish has detrimental effects on the cotton substrate.

4.1 Statistical analysis

The analysis work was done using the Statistical Packages for the Social Sciences SPSSx program (SPSS, 1983) in operation at the University of Alberta, Edmonton, Alberta, Canada. The results of the program runs are presented in Appendix IV. A oneway analysis of variance to test for a significant difference among means ($\alpha=0.01$) was performed on measurements of the dependent variables dimensional change, tensile strength, pH and colour change by the independent variable, treatment. In addition, Duncan's Multiple Range Test was used to determine which pairs of treatments were significantly different at the 0.01 level and which means formed a homogeneous subset. Homogeneous subsets are subsets of groups whose highest and lowest means do not differ by more than the shortest significant range for a subset of that size. In this study, therefore, means falling into a homogeneous subset did not differ significantly at the 0.01 level. A stringent α level was selected in order to be assured of a 99% certainty that the results had not occurred by chance because it was considered that even minor changes could be important to the aesthetic and conservation requirements for valuable art works.

4.2 Test results

4.2.1 Dimensional change

The changes in dimension of the cotton test fabric as a result of experimental treatment are recorded in Table 4.1. The results of the statistical analysis of this data are presented in Tables IV.1, IV.2, IV.3, and IV.4 in Appendix IV.

It can be seen from these results that shrinkage occurred when the cotton fabric was immersed in water. This is a normal occurrence and to be expected in fabrics that have not been stabilized. This type of shrinkage is known as relaxation shrinkage and results from a release of the tensions introduced into fibers and yarns during manufacture of the fabric. The effect of the fire retardant treatments on fabric dimensions varied but it was found that they modified the shrinkage effect of the water. This finding is difficult to explain because the release of tensions by immersion in water would occur regardless of the added chemicals. As the fabric dries the fire retardant finish may crystallize within the fabric structure. It is possible that the chemical compounds present in some way interfere with the normal repositioning of hydrogen bonds that occurs during relaxation shrinkage.

The results of the oneway analysis of variance indicate that there is a significant difference between the mean changes in dimension as a result of the experimental treatment. As can be seen in Table 4.1 the mean dimensional change for the borax:boric acid:diammonium phosphate (7:3:5) treatment is slightly lower than that for water alone but the two form a homogeneous subset in both warp and weft directions as determined by Duncan's Multiple Range Test and are therefore not significantly different. X-12 and FlameGard DSH exhibited similar behavior in this test, and these two treatments formed an homogeneous subset in both warp and weft directions.

The % changes in dimensions are relatively low. Shrinkages of 1.4 - 0.6% in the warp direction and 0.8% - zero in the weft direction are considerably less than the 3% maximum normally acceptable after fabrics such as draperies are cleaned (ASTM, 1983). Where a fiber art work is concerned, however, the dimensional change which the artist considers acceptable will vary depending upon the scale of the work and the method of installation. Should a fiber piece be installed in a free hanging [illegible] small changes in dimension would not be noticeable. If, however, the art work is designed for a specific archi[illegible]ation and, moreover, is

Table 4.1 Dimensional change of cotton treated with fire retardant finishes[1]

	warp		weft	
Treatment	change(cm)	% of original	change(cm)	% of original
water	-0.35	-1.4	-0.20	-0.8
B/BA/DAP[2]	-0.28	-1.1	-0.17	-0.7
X-12	-0.17	-0.7	0.0	0.0
DSH[3]	-0.15	-0.6	0.0	0.0

[1] mean of 6 measurements
[2] borax:boric acid:diammonium phosphate (7:3:5)
[3] FlameGard DSH

NB: a negative value indicates shrinkage

designed to be attached to the wall on all sides, dimensional change after treatment with a fire retardant finish could be crucial. The problem could be further exacerbated if a very large scale piece is involved as even a 1% change could amount to many centimetres difference in size.

Preshrinkage of the cotton will eliminate the problem of relaxation shrinkage caused by immersion in water, but without further testing it would appear that a fire retardant finish might modify the effect of the preshrinkage. This aspect does not appear to have been investigated in research into the effects of fire retardant treatments.

It should be restated at this point that in accordance with Method 25.1-1977 (CGSB,1977) the dimensions were recorded at standard conditions of 65 ± 2% RH and 21 ± 2 C. These conditions, particularly the relative humidity, are not typical of atmospheric conditions in many parts of the country. Therefore, dimensional changes will vary not only with the type of fire retardant treatment being used, but also with prevailing relative humidity. Cotton fibers can absorb moisture from the air causing them to swell and become rounder in cross section. This results in a contraction of the fabric structure, and consequently smaller dimensions.

For the artist who is working on a commission that may be installed in a different location than it is constructed, the potential for a change in dimensions as a result of different conditions should not be overlooked. It should also be noted that dimensional changes can occur whether or not a fire retardant treatment is used.

It is clear from the results of these tests that dimensional change due to fire retardant treatments requires consideration in the planning stages of a fiber commission. Pretesting is the only sure means of evaluating potential changes in dimension of a given art work when a fire retardant treatment is planned.

4.2.2 Fabric tensile strength

"Breaking strength is considered the most generally useful property of a textile material because it is indicative of the inherent quality of the textile and because it provides a useful measure for following changes or detecting damage resulting from chemical, physical, mechanical or microbiological effects in processing or in use" (Warner, 1966, p.1102). Mechanical testing for evidence of fiber degradation requires a substantial decrease in polymer chain length before an observable change occurs. Therefore, when a decrease in tensile strength is observed it is indicative of considerable destruction of the glycosidic linkages of the cellulose polymer chain.

The tensile strength of the treated cotton test fabric both before and after accelerated aging is recorded in Table 4.2.1 as mean breaking force (newtons) plus the 95% confidence interval. The percent strength retained by the fire retardant treated cotton, before and after accelerated aging, is presented in Table 4.2.2. The first column "unaged, % of control" represents the change in the tensile strength resulting from application of the finish. The breaking force of treated specimens before aging is expressed as a percent of the water treated control fabric before aging. The second column "aged, % of unaged" represents the percent strength retained when specimens were subjected to accelerated aging in an Atlas Weatherometer with carbon arc lamp. The breaking force of the aged specimens is expressed as a percent of the breaking force of the unaged specimens. The unaged fabrics were tested four months after treatment and the aged fabrics three months after completion of the accelerated aging process. Statistical analysis results are recorded in Tables IV.5, IV.6, IV.7 and IV.8 in Appendix IV.

In the unaged samples a oneway analysis of variance indicated a significant difference among the tensile strength of treated specimens. Duncan's Multiple Range Test showed that

Table 4.2.1 Mean breaking force(newtons) of fire retardant treated cotton before and after aging[1]

Treatment	Unaged	Aged
water	156.4 ± 4.5[2]	99.2 ± 2.9
B/BA/DAP[3]	153.7 ± 2.9	79.4 ± 2.1
X-12	159.4 ± 4.1	22.6 ± 1.7
DSH[4]	166.2 ± 3.2	23.6 ± 1.3

[1] mean of 10 breaks
[2] mean ± 95% confidence interval
[3] borax:boric acid:diammonium phosphate (7:3:5)
[4] FlameGard DSH

Table 4.2.2 Percent strength retained by fire retardant treated cotton before and after aging

Treatment	Unaged, % of control	Aged, % of unaged
water(control)	100.0	63.4
B/BA/DAP[1]	98.3	51.7
X-12	101.9	14.2
DSH[2]	106.3	14.2

[1] borax:boric acid:diammonium phosphate (7:3:5)
[2] FlameGard DSH

water, borax:boric acid:diammonium phosphate (7:3:5), and X-12 formed a homogeneous subset, therefore, the tensile strength of these fabrics was not significantly different. Fabric treated with FlameGard DSH was shown to be significantly different from fabric given the other three experimental treatments. It exhibited a 6.3% increase in strength compared to the control fabric. An increase in tensile strength after treating cotton with fire retardant finishes has been noted by other researchers (Marsh, 1957). Cotton fibers typically exhibit an increase of 10 - 20% in strength when wet (Joseph, 1977). It seems possible, therefore, that the increased moisture content of the fabric attributed to the hygroscopic nature of the fire retardant finish could have imparted this strength increase to the cotton fabric treated with FlameGard DSH. The presence of the fire retardant finish may also affect the mobility of the fibers and by introducing friction may impart a slight increase in tensile strength. Water soluble fire retardant finishes, in general, have been found to lower tensile strength by 5 - 15%, although this strength loss is not considered excessive (Little, 1947; Lyons, 1976).

A oneway analysis of variance of the data from the aged specimens indicated a significant difference among the tensile strength of these samples. Duncan's Multiple Range Test indicated that fabrics treated with X-12 and FlameGard DSH formed a homogeneous subset. Both treatments caused a substantial loss of tensile strength in the cotton fabric after aging, the treated fabrics retaining only 14.2% of their strength before aging. Agents containing ammonium sulfate have been found to cause considerable loss of strength in response to heat aging (Marsh, 1957). FlameGard DSH was found to contain sulfate in the chemical analysis. Fabric treated with borax:boric acid:diammonium phosphate (7:3:5) was significantly different in tensile strength after aging compared to the X-12 and FlameGard DSH treated specimens. In addition, the strength of aged fabrics treated with all three fire retardant treatments were significantly different from the water control treatment. The borax:boric acid:diammonium phosphate (7:3:5) treated fabric exhibited the best strength retention. It retained over 50% of its strength before aging. Borax and ammonium phosphate have been found to have the least destructive action of the inorganic fire retardant agents (Marsh, 1957). However, there is some question as to whether this treatment survived the conditions of accelerated aging for any length of time. Loss of this retardant is known to occur through volatilization (Little, 1947). In borax-boric acid mixtures that have added ammonium phosphate the loss of ammonia causes the formation of an acid phosphate which accelerates the sublimation of boric acid and also causes an interaction between borax and the phosphate forming additional amounts of volatile boric acid (Little, 1947).

Sunlight and water are the main elements that destroy fire retardant agents (Drake & Reeves, 1971). . Whilst the level of moisture in the Weatherometer accelerated aging chamber may not have been high enough to cause leaching it seems possible the that high level of radiant energy to which the samples were exposed could destroy the borax:boric acid:diammonium phosphate (7:3:5) treatment during the cycle of aging. Even without aggravated conditions this particular fire retardant treatment been found, in previous experiments, to have lost practically all its fire retardancy characteristics after one year's exposure to normal room conditions (Little, 1947).

The results of the tensile tests indicate that there is a strength loss in the control fabric after aging of 36.6%. This means that without any chemicals present on the fabric degradation has occurred, as a result of the action of heat and light, causing a loss of over one third of fabric strength. The fabric treated with borax:boric acid:diammonium phosphate (7:3:5), by comparison, lost almost one half of its tensile strength during accelerated aging. The fact that the borax:boric acid:diammonium phosphate (7:3:5) treated fabric lost more strength than the untreated control fabric can, therefore, be attributed to the effect of the finishing treatment degradation products.

According to Little (1947), it is the fabrics that retain some or most of their fire resistance under accelerated aging conditions that are most severely impaired with regard to fabric strength. Although testing for retained fire resistance was not a part of this study, such a finding could account for the results obtained from the tensile tests of X-12 and FlameGard DSH treated fabric. Both treatments contributed to over an 85% loss of strength in the fabrics after exposure to the conditions of accelerated aging. Both treatments fall into the generic category of inorganic ammonium salts. Ammonium phosphate and ammonium sulfate have been found to be the most common constituents of such mixtures. These ammonium salts are unstable and apt to become acidic resulting in damage to the cloth (Marsh, 1957). Although the exact composition of the two commercial reagents is not known, it is thought that X-12 is an ammonium sulfamate-phosphate combination. Mixtures such as this have been found to retain their effectiveness under accelerated aging procedures similar to those used in the present study and to show an excessive accompanying loss of tensile strength (Little, 1947).

The findings of this test appear to have grave consequences for fiber art. The relationship of the accelerated aging procedure to normal use conditions of fiber art work, however, is not definable. The conditions of 60-65 C and continuous high intensity ultra violet light would never be encountered under normal conditions and it would take an enormous length of time, if ever, for the effect to be achieved in a cumulative manner. The results do indicate, however, that heat and light interact with the chemical system formed by the cellulose and the fire retardant finish in a more aggressive manner than occurs when a fire retardant finish is not present on the cellulose; therefore, the degradation occurring in cotton fabric as a result of environmental conditions is greater when a fire retardant finish is present.

4.2.3 Fabric pH

The reason for the concern of the textile conservator about the pH level of cellulosic textiles is because of the ability of acids to promote hydrolysis of the 1,4 glycosidic links of the cellulose polymer chain. The increase in chain ends resulting from these broken links leaves the fabric in a reactive state and, therefore, more vulnerable to further degradation. Hydrolysis leads to a reduction in DP and a concomitant reduction in fiber tensile strength with the ultimate result being the disintegration of the fabric structure. However, other factors such as light also play a role in fiber degradation. Interaction between different degradative factors precludes direct correlation between specific pH levels and amount of fiber destruction. Nor can the pH value obtained in the testing be taken as definitive because of the potential for contamination from sources such as CO_2 in the air. However, changes in the hydrogen ion concentration in aqueous solution(pH) of the fabric do provide a means of assessing modifications in the level of acidity of cotton fabric. pH is the negative logarithm of the hydrogen ion concentration. The decrease from a neutral level of pH 7 to pH 6, therefore, means a tenfold increase in acidity. A decrease from pH 7 to pH 5 means a one hundredfold increase in acidity. The pH measurements of the treated fabrics, before and after aging, are presented in Table 4.3, and the results of the statistical analysis can be seen in Tables IV.9, IV.10, IV.11 and IV.12 in Appendix IV.

Table 4.3 The effect of fire retardant finishes and accelerated aging on the pH of cotton fabric[1]

Treatment	Unaged[2]	Aged[3]
water (7.6)	7.2	6.2
B/BA/DAP[4] (8.4)	7.3	6.1
X-12 (6.7)	6.3	3.8
DSH[5] (8.2)	5.5	4.1

[1] pH of treatment solution in parentheses
[2] mean of 7 determinations
[3] mean of 2 determinations
[4] borax:boric acid:diammonium phosphate (7:3:5)
[5] FlameGard DSH

A oneway analysis of variance for the unaged fabrics indicates a significant difference among pH means. The fabrics treated with water and borax:boric acid:diammonium phosphate (7:3:5) both remained close to neutral having a pH of 7.2 and 7.3 respectively. These two treatments formed a homogeneous subset in Duncan's Multiple Range Test and therefore, are not significantly different. The X-12 treated fabric was slightly acidic with a pH of 6.3 and FlameGard DSH was the most acidic with a pH of 5.5. These two treatments were significantly different from water and borax:boric acid:diammonium phosphate (7:3:5) and from each other. None of the levels of acidity found in this test are considered aggressive enough to be damaging to the cellulose fibers especially in the short term.

A oneway analysis of variance of the data from the aged samples indicated a significant difference among mean pH readings. As with the unaged fabrics, Duncan's Multiple Range Test showed that water and the borax:boric acid:diammonium phosphate (7:3:5) treatment, with mean pH values of 6.2 and 6.1 respectively, were not significantly different. FlameGard DSH treated fabric with a pH of 4.1 and X-12 treated fabric with a pH of 3.8 were both significantly different from the water and borax:boric acid:diammonium phosphate (7:3:5) treatments and from each other.

The relationship of the pH of the treated, unaged fabric to the pH of the treatment preparation was not as expected in all cases. The control fabric and that treated with X-12 exhibited a difference of only 0.4 pH units between the pH of the treatment solution and the pH of the treated fabric. Because the pH values are close to neutral this difference is not a concern. The pH of the borax:boric acid:diammonium phosphate (7:3:5) treatment was slightly basic (8.4) and that of the treated fabric was close to neutral at 7.3. A mixture of borax and boric acid has been promoted as a buffering agent in the deacidification of paper (Daniels, 1980). It is said to have a stable pH and be capable of neutralizing residual acids. When diammonium phosphate, which is basic but known to lose ammonia through volatilization, is added to the mixture it becomes less stable. In the borax:boric acid:diammonium phosphate (7:3:5) mixture, phosphate remaining after volatilization of the ammonia can react with borax to create boric acid (Little, 1947). These two factors could account for the drop in pH from 8.4 for the treatment solution to 7.3 for the treated fabric.

The major difference between treatment pH and fabric pH occurred with the FlameGard DSH treatment. Whilst the pH of the treatment solution was 8.2 the pH of the treated fabric was found to be 5.5. It is not possible to explain this result due to a lack of knowledge of the composition of the proprietary mixture. Technical information accompanying the product indicated that an ammonia odor was to be expected on use. This loss of ammonia could be one reason for the noted decrease in the pH of the treated fabric.

After the treated fabrics were subject to accelerated aging in an Atlas weatherometer the pH of the fabric was lowered as was to be expected. Cotton fabrics tend to become acidic with age, and fire retardant finishes typically become acidic on breakdown induced by heat and light. The increased level of acidity, however, was only marked for the X-12 treated fabric. The change from 6.3 to 3.8 represents well over a hundredfold increase in the hydrogen ion concentration. There is a dearth of research on this particular aspect of fire retardant treated cotton. However, one study did indicate that a fire retardant finish containing diammonium phosphate, but no borax or boric acid, did not have any appreciable effect on fabric pH after heat aging (Williams, 1974). The levels of acidity found are, in general, not considered aggressive for short term purposes (Leene, 1972); but it must be realised that any long term exposure of cellulosic fibers to even low levels of acidity can only have deleterious effects on the fiber structure however minimal it may be. Whilst avoidance of acidic properties in cellulosic fiber art is highly desirable minor levels of acidity can be tolerated.

It is obvious from the results of the testing that the selection of a fire retardant finish on the basis of the pH level of the treatment mixture will not automatically provide a similar level of pH within the fabric; and it is to be expected that the level of acidity of the fabric will increase over time with exposure to heat and light.

4.2.4 Colour change

The problem of colour change in textiles treated with a fire retardant finish can give the textile conservator two reasons for concern. In cotton textiles a colour change can cause not only aesthetic problems, but can also be indicative of fiber degradation. Natural, undyed cotton can be bleached by sunlight. The radiant energy absorbed also results in photo-sensitized degradation. The amount of degradation occurring will vary with the impurities in the cellulose; the more impure the cellulose the more degradation results. Yellowing or darkening of cotton is a visual indication that the cellulose molecules have been oxidized. Whilst oxidation does not cause fracturing of the cellulose polymer chain it leaves the fiber in a more reactive state and vulnerable to hydrolysis which does result in a reduced DP and loss of fiber strength.

Undyed cotton: Colour change as a result of the application of a fire retardant finish to undyed cotton and as a result of the accelerated aging on the treated cotton is shown in Table 4.4.1. Changes in colour have been measured and calculated by comparison to the water treated control fabric. In order to give meaning to the numerical values for colour change an AATCC Gray Scale rating has also been included (AATCC, 1982/3). Verbal descriptions of the Gray Scale ratings appear in Table 4.4.2. For the purposes of this study a Gray scale rating of 4 has been selected as the maximum level of colour [illegible] acceptable (ASTM,1983). Statistical analyses of results are presented [illegible] and IV.14 in Appendix IV.

A oneway analysis of variance indicated a significant difference among the mean colour changes observed in the undyed fabrics. Duncan's Multiple Range Test showed that the colours of the aged fabrics treated with X-12 and FlameGard DSH were significantly different from the unaged samples and the aged samples treated with water and borax:boric acid:diammonium phosphate (7:3:5). Homogeneous subsets could not be obtained because of the unbalanced design for this data.

Undyed fabrics treated with fire retardant finishes exhibited no noticeable colour change in the unaged state. Upon being subjected to accelerated aging the treated fabrics varied in their response to the conditions of the aging process. The fabrics treated with X-12 and FlameGard DSH became tan in colour possibly due to the interaction of an agent in the finish with the intense light of the carbon arc lamp. Lai et al (1977), investigating the effects of chlorine in fire suppression recorded a tan coloured substrate upon chlorination of cellulose. Chemical analysis of FlameGard DSH indicated that it contained a lot of chloride, although none was found in X-12. A further example of colour change on a natural coloured fiber is provided by Platus (1977) who found that an area contaminated by mold turned "toast brown" when treated with a fire retardant finish.

The water and borax:boric acid:diammonium phosphate (7:3:5) treated fabrics both became whiter as a result of accelerated aging, the change being somewhat noticeable compared to the unaged control fabric. The use of sunlight is a traditional method of bleaching cellulosics. Radiation from a carbon arc lamp is similar but not identical to visible light.

Table 4.4.1 The effect of fire retardant treatment and accelerated aging on the colour of undyed cotton[1]

Treatment	State	Colour change ΔE (CIELAB units)	AATCC rating[2]
water	unaged	0.0	5
	aged	2.52	3-4
B/BA/DAP[3]	unaged	0.99	4-5
	aged	2.29	3-4
X-12	unaged	0.91	4-5
	aged	10.54	1-2
DSH[4]	unaged	0.60	4-5
	aged	13.20	1

[1] calculations based on the mean of 5 readings for the unaged samples; 8 readings for the aged borax:boric acid:diammonium phosphate (7:3:5) sample; and 10 readings for the X-12 and FlameGard DSH aged samples
[2] see Table 4.4.2
[3] borax:boric acid:diammonium phosphate (7:3:5)
[4] FlameGard DSH

Table 4.4.2 Relationship of colour change in CIELAB units to AATCC Gray Scale ratings with verbal description

Colour change CIELAB units	AATCC Gray Scale rating	Verbal description
0.0 ± 0.2	5	negligible or no change
0.8 ± 0.2	4-5	
1.7 ± 0.3	4	slightly changed
2.5 ± 0.35	3-4	
3.4 ± 0.4	3	noticeably changed
4.8 ± 0.5	2-3	
6.8 ± 0.6	2	considerably changed
9.6 ± 0.7	1-2	
13.6 ± 1.0	1	much changed

Thus, it is possible that the colour changes seen during accelerated aging would not occur during natural aging.

Dyed cotton: The colour changes which occurred when the swatches dyed with Procion® fiber reactive dyes were treated with fire retardant agents are reported in Table 4.4.3. The results of the statistical analysis of this data can be found in Tables IV.15 through IV.26 in Appendix IV. It can be seen that no particular treatment caused a marked colour change in all dyed fabrics. The borax:boric acid:diammonium phosphate (7:3:5) treatment, for example, caused a noticeable change in the red, blue and orange swatches. X-12 caused a noticeable change in the red and blue samples. FlameGard DSH caused a noticeable change in the red, yellow, blue and orange samples.

Statistical analysis was performed on the colour change data for the dyed samples individually by colour. A oneway analysis of variance indicated a significant difference among mean colour changes for each colour. Duncan's Multiple Range Test results are summarized below:

Red: FlameGard DSH and X-12 formed a homogeneous subset and X-12 also formed a homogeneous subset with borax:boric acid:diammonium phosphate (7:3:5); all treatments were significantly different from the water control treatment.

Yellow: Each treatment formed a separate subset, therefore, all treatments were significantly different from each other.

Blue: FlameGard DSH and X-12 formed a homogeneous subset and were significantly different from the water treated control and from the borax:boric acid:diammonium phosphate (7:3:5) treatment.

Green: Each treatment formed a separate subset, therefore, all treatments were significantly different from each other.

Orange: Each treatment formed a separate subset, therefore, all treatments were significantly different from each other.

Purple: FlameGard DSH and borax:boric acid:diammonium phosphate (7:3:5) formed a homogeneous subset and were significantly different from both the X-12 and water control treatments.

In this research the testing of fire retardant finishes on dyed cotton swatches was limited in scope and provides the artist with only a general indication of the potential for colour

Table 4.4.3 The effect of fire retardant treatment on the colour of cotton dyed with Procion® fiber reactive dyes.

Colour	Treatment	Colour change ΔE (CIELAB units)[1]	AATCC Gray Scale rating
Red	B/BA/DAP[2]	3.02	3
	X-12	2.82	3-4
	DSH[3]	2.70	3-4
Yellow	B/BA/DAP	0.74	4-5
	X-12	1.72	4
	DSH	2.60	3-4
Blue	B/BA/DAP	2.95	3-4
	X-12	2.26	3-4
	DSH	2.33	3-4
Green	B/BA/DAP	0.92	4-5
	X-12	1.28	4
	DSH	2.06	4
Orange	B/BA/DAP	3.17	3
	X-12	1.44	4
	DSH	2.46	3-4
Purple	B/BA/DAP	1.53	4
	X-12	1.98	4
	DSH	1.39	4

[1] mean of 5 readings
[2] borax:boric acid:diammonium phosphate (7:3:5)
[3] FlameGard DSH

change. The results obtained can only be considered applicable to the specific dyes and fire retardant chemicals used in the study. Hunnings (1976) in *Fire retardancy and the interior designer* (1976), graphically illustrates a problem he encountered with colour change caused by a fire retardant finish; the design concept was totally altered and a significant cost overrun was encountered. Platus (1977) also cautions the fiber artist of this potential problem.

It should be emphasized again that pre-testing is essential when a fiber art work must be treated with a fire retardant finish. It is the only way to obtain a valid prognosis for a particular project.

4.2.5 Fabric stiffness

Fabric stiffness is a measurable component of the fabric properties of hand and drape. Hand, particularly, is a property that relies to a great extent on descriptive, subjective evaluation, whilst drape is more measurable in terms of bending length and flexural rigidity. Measurements of flexural rigidity have been shown to give excellent correlation with subjective evaluations obtained by the handling of fabric samples. A 10% difference in flexural rigidity is reported as being the minimum level capable of being detected subjectively (ASTM, 1983). A negative change indicates that the fabric has lost stiffness, that is, it has become limper. A positive change in flexural rigidity indicates that the fabric has become stiffer.

Flexural rigidity was measured under both standard and ambient conditions because the standard relative humidity of 65 ± 2% is considerably higher than that found in many areas of the country. Measurements of flexural rigidity and % change in fabric stiffness are recorded in Table 4.5.

Under standard conditions the stiffness of the fabrics was considerably reduced by the fire retardant treatments when compared to the original fabric. The greatest change occurred in the fabric treated with FlameGard DSH which was 38.1% less stiff than the original fabric. Simple treatment with water, however, accounted for a loss of 12.3% of stiffness, so that the fire retardant finish accounted for approximately 25% of the loss of stiffness. The borax:boric acid:diammonium phosphate (7:3:5) and X-12 treated fabrics exhibited a 16% and 12.9% reduction in stiffness respectively compared to the control. The hygroscopic nature of the fire retardant finishes is thought to have contributed to the high loss of stiffness under the standard relative humidity of 65 ± 2%.

Under an ambient relative humidity of 21% the change in stiffness compared to the water treated control fabric was reversed for the borax:boric acid:diammonium phosphate (7:3:5) and X-12 treatments. The fabrics treated with these two fire retardant agents exhibited an increase in stiffness of 20.8% and 12.6% respectively. It should be noted, however, that in measuring the stiffness of the control fabric under ambient conditions an

Table 4.5 The effect of fire retardant treatment and relative humidity on the stiffness of cotton fabric[1]

Treatment	Conditions[2]	Flexural rigidity (mg/cm)	% change from original	% change from control
original	standard	118.946	-	-
	ambient	416.550	-	-
water (control)	standard	104.260	-12.3	-
	ambient	335.153	-19.5	-
B/BA/DAP[3]	standard	87.576	-26.4	- 16.0
	ambient	404.929	- 2.8	+ 20.8
X-12	standard	90.831	-23.6	- 12.9
	ambient	377.426	- 9.4	+ 12.6
DSH[4]	standard	73.575	-38.1	- 29.4
	ambient	332.864	-20.1	- 0.7

[1] calculated from 16 measurements of bending length in both warp and weft direction
[2] standard conditions: 65±2% RH; ambient conditions: 21% RH
[3] borax:boric acid:diammonium phosphate (7:3:5)
[4] FlameGard DSH

inconsistency exists. The numerical values indicate a higher loss of stiffness, which suggests that the fabric is limper under ambient conditions (21% RH) than under standard conditions (65±2% RH). This result is difficult to justify, but one possible explanation is that a higher degree of distortion (twisting) occurred in the leading edge of the sample when stiffness was measured at 21% RH, which may have increased the error in bending length measurement with a fabric lower in moisture content. Under ambient conditions the fabric treated with FlameGard DSH exhibited a similar degree of stiffness to the control fabric.

Although a controlled, scientific test for fabric hand was not conducted, subjective evaluation by three co-workers was done in the laboratory at 21% RH. The fabric treated with borax:boric acid:diammonium phosphate (7:3:5) was judged to have a "chalky", crispy hand and to be the stiffest of the experimental treatments. The X-12 treated fabric also felt slightly crispy and stiff but less so than the borax:boric acid:diammonium phosphate (7:3:5) treated fabric. The fabric treated with FlameGard DSH felt quite similar to the water treated control but was said to have a slick or glossy feel to it.

There has been considerable emphasis on hand and drape of fabrics treated with fire retardant finishes in past research because frequently the finishes were required for use on clothing. For example, Little (1947) reported that borate mixtures impart a slight harshness to the cloth, whereas the sulfamates and their mixtures function as softening agents producing a slightly softer finish. Marsh (1957) also supports this finding, stating [illegible] a special feature of ammonium sulfamate is its lack of adverse effects on the hand of the cloth. Lewin and Sello (1975) state that the softening effect is independent of humidity. If X-12 is a sulfamate/phosphate preparation it appears the phosphate may be contributing to the stiffening. Lindsay (1966) agrees that the borate mixtures leave 'goods' with a stiff hand, but suggests that ammonium sulfamate does affect hand to some extent.

For fiber art works a change in hand, which implies tactile sensations, will rarely be a factor to consider in the selection of a fire retardant finish. The visual impact derived from draping qualities, in contrast, could be very important depending upon the construction technique involved and the visual image the artist wishes to project. A two dimensional piece that is mounted on a wall is unlikely to be affected visually by changes in stiffness. However, if a hanging banner were to be treated with a fire retardant finish and became either boardy or very limp its visual qualities would be impaired. For a three dimensional piece which relies upon fabric manipulation for its aesthetic impact a loss of stiffness could alter the artistic concept. The importance of changes in fabric stiffness, therefore, will vary depending upon the art work involved and the artist's requirements of aesthetics and structure.

In addition to the specific test results which have been discussed, other effects from the use of fire retardant agents on the cotton substrate have been observed. Samples of fabric treated with the three different finishes were stapled to paper and hung in the conditioning room to observe the effects of continued exposure to standard conditions of 65% ± 2% RH and 21 ± 2 C. As has been previously mentioned, the borax:boric acid:diammonium phosphate (7:3:5) treatment leached out under conditions of high humidity. Crystals appeared on the surface of the fabric within two days. Within the space of three months the surface of the fabric was free of crystals, thus it is possible that the finish had completely volatilized away.

Approximately two months after being placed in the conditioning room the samples treated with FlameGard DSH were observed to have caused corrosion of the staples. Within three months, that is, a month after the corrosion was noticed the fabric around the staples was heavily stained a dark yellow colour. Ammonium sulfate has been found to accelerate the corrosion of steel and other metals compared to the corrosion occurring when no finish is present (Lyons, 1970). The fiber artist should note this effect, which is also mentioned by Platus (1977), when considering the use of metal support mechanisms in conjunction with a fire retardant finish. The humidity level had no observable effect on the X-12 treated fabric.

5. SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

5.1 Summary

The major objective of this research was to identify suitable fire retardant agents for use on art works in the fiber medium. To be judged suitable an agent must first of all impart the required level of fire resistance to enable the treated fabric to pass the vertical flame test. Secondly, there must be an absence of deleterious effects on fiber properties and thirdly, an absence of aesthetic changes in the art work. To evaluate the latter two requirements, treated fabrics were tested for changes in dimension, colour, stiffness, tensile strength and pH after treatment; and for changes in colour, tensile strength and pH after treatment and accelerated aging in an Atlas weatherometer.

Textile conservators charged with the care and treatment of contemporary textile art are concerned about the application of fire retardant chemicals to valuable fiber works. The results of the tests performed in this study indicate that such concern is justified. The short term changes observed are, in the main, of minor importance except in very specific instances: for example, a change in stiffness could affect the appearance of a piece relying on drapeability qualities. There is no doubt, however, that in the long term the presence of a fire retardant agent on a cellulosic substrate tends to magnify the effect of natural degradation processes.

FlameGard DSH was the easiest treatment to prepare and apply and the treated fabric passed the flame test most easily of the three treatments in spite of the fact that it had only about two thirds of the add-on of the other two. It caused negligible dimensional change and negligible colour change on undyed, unaged fabrics. When applied to dyed fabrics it caused a noticeable change in the red, yellow, blue and orange swatches. Treated samples were quite limp at 65% RH but the same as the water treated control fabric at 21% RH. Tensile strength of the treated fabric before aging was increased. It did cause a totally unacceptable colour change, however, and a substantial loss of tensile strength in the undyed, aged cotton. The pH level of the treated fabric before aging was 5.5, considerably less than the pH of the treatment mixture, and lower than that considered desirable for cellulosic textiles. After aging the pH was lowered further, a fact reflected in the weakened state of the cotton fabric.

The other proprietary reagent, X-12, required a much higher add-on (15.2%) to achieve the desired level of fire resistance than did FlameGard DSH. Being in powdered form it was less easily prepared than FlameGard DSH but, once dissolved, application was accomplished just as easily. In many respects it had similar effects on the cotton to FlameGard DSH. Negligible dimensional change occurred and a slight increase in tensile strength before aging was followed by a substantial loss of tensile strength after aging. X-12 and FlameGard DSH caused the same colour change of the dyed swatches in all cases except the fabrics dyed orange and yellow. Fabrics treated with X-12 and FlameGard DSH had slightly different pH values , although the same general trend was seen. X-12 was less acidic (pH 6.3) on the fabric before aging and more acidic (pH 3.8) after aging than FlameGard DSH. A major difference between these two reagents occurred in the stiffness test with the X-12 treated fabric exhibiting greater stiffness under standard conditions (65% RH) and ambient conditions (21% RH).

The borax:boric acid:diammonium phosphate (7:3:5) treatment also required as high an add-on (15%) as the X-12 treatment. This mixture required time and care in the dissolving of the chemicals, and the fabric required three treatments before sufficient add-on was attained to enable it to pass the vertical flame test. Of the three fire retardant finishes it exhibited the most acceptable levels of pH before and after aging. It was the only finish that did not darken the fabric upon aging but it caused more noticeable colour change in two of the dyed swatches, namely red and orange, than did X-12 and FlameGard DSH. It had a greater effect on dimensional change, having shrinkage of over 1% in the warp direction and 0.7% in the weft direction, behaving in a similar manner to the water treated control fabric. Tensile strength of the fabric after treatment with borax:boric acid:diammonium phosphate (7:3:5) was minimally lower (98.3% of water treated control), but this treatment gave by far the best results in tensile tests after aging. After almost 500 hours of exposure to a temperature of 60-65 C and 20-35% RH with continuous carbon arc light it retained over 50% of its after treatment strength, compared to the 14% retained by the samples treated with X-12 and FlameGard DSH. Its effect on fabric stiffness was the most undesirable of the three finishing treatments examined leaving the fabric feeling "chalky" and "boardy" at low relative humidity.

5.2 Conclusions

From a conservation perspective the three fire retardant finishes examined for this study all possess some shortcomings. There were two major problems with the borax:boric acid:diammonium phosphate (7:3:5) treatment; firstly, the difficulty of obtaining an effective level of add-on necessitated repeated applications which are more stressful to textile fibers than a single application; and secondl. .he the chemicals appeared to leach out of the fabric with ease at 65% RH. Previous research attests to the short life expectancy of this finish even under normal room conditions (Little, 1947).

In this study the chemical agents were applied to the test fabric as it came from the bolt. It was not washed before treatment because it was suspected that few fiber artists would prepare yarns or fabrics by washing before use. A pre-wash, however, would allow for better pick-up of the fire retardant chemicals and the desired level of add-on would likely be achieved without repeated immersions. A wash would also act to pre-shrink the yarns or fabric. If it is suspected that the fire resistance properties of the borax:boric acid:diammonium phosphate (7:3:5) are being lost a maintenance program should be established for an art work treated with this finish. It should be cleaned and retreated on a frequent basis so that the intent of the fire safety regulations is not defeated, and so that damaging products from the degradation of the fire retardant finish are removed. This approach is only advocated on the basis that all other aspects of this treatment are acceptable.

The two proprietary reagents, X-12 and FlameGard DSH, had similar effects on fabric properties and aesthetic changes. Ease of preparation and lower add-on make FlameGard DSH a better choice than X-12; however, given the aggressive effects displayed by each reagent in the aging process, neither treatment, at this stage, can be advocated for long term use on valuable textiles. As an immediate and short term solution to the problem of having to treat an existing piece in order to comply with regulations either treatment could be considered for use with the proviso that pre-testing be done to determine suitability for a specific application. Should such an approach be considered necessary it would be incumbent upon the artist or conservator to establish a monitoring system whereby signs of degradation could be observed in the early stages. This course of action would allow an opportunity to clean the piece to remove the finishing agents and degradation products. The situation would have to be reappraised at that point to determine whether retreatment should take place or the art work removed.

It must be stated that the conclusions being drawn here are based on the results of the specific tests performed before and after accelerated aging and such conclusions are appropriate only for the chemical reagents used in the study. The advice given by Drake, Perkins and Reeves (1973, p.25) is still appropriate: "At this state of the art, no one finish is applicable to all types and weights of fabric, nor for all end uses. In selecting the best treatment for a specific application, one must consider fabric construction, properties affected by the finish, end-use, chemical and processing costs, and the availability of materials". The fiber artist who *has* to apply a fire retardant finish in order to install a work of art must, therefore, evaluate any proposed treatment by the criteria above and on the basis of which changes can be most readily accepted. Whether the fire retardant treatment is applied by a commercial company or whether the artist applies the finish personally, it can be done either before construction of the art work by treating the materials to be used, or after construction by treatment of the finished piece. There are advantages and disadvantages to both approaches. These are well outlined by Platus (1977) who has learned through experience the pitfalls of both. Pretesting of proposed treatments cannot be stressed strongly enough. Planning the treatment as part of the project rather than allowing it to be an afterthought allows the opportunity for pretesting and will eliminate many of the pitfalls.

A major point in the overall issue of fire retardancy that must be emphasized is the fact that fire resistance properties in a fiber art work can be achieved without the use of finishing treatments. As has been mentioned in section 2.5 several factors influence the flammability potential of a textile construction. The use of inherently non-flammable fibers and a high density structure are two methods of construction that can be employed to produce an art work capable of passing the vertical flame test. This may constitute somewhat of a dilemma to the fiber artist. It may be difficult or impossible to achieve the desired effect with an inherently non-flammable construction, or the required materials simply may not be available. However, at some point the fiber artist will most likely have to acknowledge the existence of fire safety codes and come to terms with the regulatory requirements of the Fire Marshall. Any piece created for a public space covered by the regulations can be required to pass the vertical flame test should it constitute more than 10% of the surface area it occupies.

In practical terms, the piece as a whole or part cannot be tested, therefore, it behooves the artist to prepare a suitably realistic model or sample which can be tested. It has been proven that the flammability behavior of a system (such as a fiber work) is *not* predictable from knowledge of the behavior of the individual components (Yeager, 1972). This means that testing of the individual components of a fiber art work will not suffice, and a composite structure identical to the finished work but smaller in scale could be required for proper evaluation.

Fire retardant finishing treatments should possibly be viewed as a last resort for fiber artists. They do have to be considered, however, for use on existing textile works some of which may be historical pieces. In the latter case the conservator's concerns gather added weight. An occurrence of improper fire retardant treatment on museum textiles was one of the factors contributing to the initiation of this research.

The indiscriminate use of fire retardant agents on valuable textiles must be avoided. When there is no alternative to treatment, care in selection of a treatment is essential. Textile conservators would do well to consider the potential damage any treatment may cause in the long term and endeavor to modify problems by a regular program of washing to remove an old and degrading finish and the application of a new one if it proves necessary. Failure to evaluate fire retardant treatments for a specific application only provides for problems in the years ahead. By then, of course, damage from the acidic residues resulting from the breakdown of the fire retardant could be extensive and expensive to treat; it may even be so great that nothing can be done and the piece is irretrievable.

This kind of scenario also has long range implications for the fiber artist. An artist's work may be popular and commissions be obtained over a short term on aesthetic capabilities alone. But in the long term other professional qualities besides creative ones are required to maintain a reputation and develop status as an artist of note. Quality of technique and the finishing of textile pieces cannot be stressed highly enough. Second only to paper in vulnerability to environmental degradation, textile fibers need knowledgeable treatment for optimum preservation.

Confronting the fire retardancy issue and dealing with it successfully can only be advantageous to the fiber artist in the long term. Commissions will be lost to artists who work in media of a non-combustible nature if those with the power to grant commissions perceive the fire retardancy of textile pieces as a barrier (Davis, 1979). Fiber works have not been readily accepted into the fine arts community. Traditionally textiles have been linked with craft and guild organizations. This association with crafts has created a resistance to their inclusion in the fine arts dicipline. Today's fiber artists have worked hard, and continue to do so, to become accepted as creators of "fine art", and to have their work accepted as equal to painting and sculpture. It would be most regrettable if the effort was negated by the failure to deal with the issue of fire retardancy.

5.3 Recommendations

Due to the conservation orientation of this study it is not possible, in light of the results obtained, to recommend unequivocally any of the reagents for use on valuable textile art pieces. It has been demonstrated by the high loss of tensile strength, the increase in acidity and the unacceptable colour change after accelerated aging that the potential for damage in the long term exists. The possibility of the damage being extensive cannot be excluded.

A major recommendation to be made is that when a piece must be treated, the fire retardant finish be considered a short term measure and provision made for removal of the finish at a specified time. Depending upon the situation the treatment could be reapplied or the textile placed in a different location not subject to fire safety regulations.

By necessity, this study has been limited in scope to three finishes readily available to fiber artists in Western Canada and it is suggested that more finishes be evaluated, particularly additional finishes that are available to fiber artists or are being used by commercial applicators. There are also many non-proprietary mixtures that could be investigated, although these tend to have been well researched in the past (Little, 1947). Because research into fire retardant finishes is ongoing, albeit at a slower pace than ten years . new finishes may appear on the market at any time and will, hopefully, be devoid of the undesirable characteristics found to date. The ideal fire retardant will be durable in nature and not affect fiber properties or aesthetics in any way; will be stable to all environmental conditions but unstable at flame temperatures; and it will be easy to apply and non-toxic.

Because the application of fire retardant finishes has been found to cause increased degradation when compared to that occurring when no finish is present on the textile, another major recommendation to be made is the necessity for educating fiber artists in the theoretical aspects of their medium. A knowledge of fiber science and textile conservation will enable the artist to use fibers, yarns, fabrics, dyes and finishes in a technically competent manner, thereby, allowing the creation and treatment of art works best able to comply with fire safety regulations with a minimum of deleterious effects on the fibers. A greater understanding of the fundamentals of fiber properties, rather than restricting aesthetic expression, could generate exploration that otherwise may not be considered. It could lead to aesthetically exciting art works that do not need a fire retardant finish in order to pass the vertical flame test. Thus, "an equilibrium between creativity and preservation" can be achieved. (Kimura, Note 1, 1984).

It is hoped that fiber artists and textile conservators will communicate and cooperate in the production and fire retardant treatment of the textile works that will find their way into the art market. Only when the aesthetically accepted works have proven they can comply with fire safety regulations and can survive over time, without damage from a fire retardant finishing treatment, will a future for fiber as an art medium be on firm ground.

BIBLIOGRAPHY

AATCC. *Technical Manual of the American Association of Textile Chemists and Colorists*, 58, 1982/3

Aenishanslin, R. Flame-retardant finishes for cellulosic fibers. *CIBA Review*, 1969, 4, 35-43.

Alvares, N.J. Ignition inhibitors for cellulosic materials. *Journal of Fire Retardant Chemistry*, 1978, 5, 42-47.

ASTM. *Annual Book of Standards*, 07.01, American Society for Testing and Materials, 1983.

ASTM. *Standard Performance Specifications for Textile Fabrics*. 1st.Edn., American Society for Testing and Materials, 1983.

Baitinger, W.F. Cellulose reactive fire retardants. *Textile Chemist and Colorist*, 1972, 4(7), 172-176.

Barker, R.H. and Hendrix, J.E. Flame retardance of cotton and other naturally occurring cellulosic polymers. *Flame Retardancy of Polymeric Materials, Vol.5*, Kuryla, W. and Papa, A., Eds., New York: Marcel Dekker Inc., 1979.

Benisek, L. Flame retardance of protein fibers. *Flame-Retardant Polymeric Materials*, Lewin, M., Atlas, S.M. and Pearce, E.M., Eds., New York, Plenum Press, 1975.

CGSB. *National Standard of Canada: Textile Test Methods*. Canadian Government Specifications Board, Ottawa, Ontario. 1977.

Chamberlain, D.L. Mechanisms of fire retardance in polymers. *Flame Retardancy of Polymeric Materials, Vol.4*, Kuryla, W. and Papa, A., Eds., New York: Marcel Dekker Inc., 1978.

Constantine, M. and Larsen, J.L. *Beyond Craft: The Art Fabric.* New York: Van Nostrand Reinhold Co., 1972.

Constantine, M. and Larsen, J.L. *The Art Fabric: Mainstream.* New York: Van Nostrand Reinhold Co., 1981.

Daniels, V. Aqueous deacidification of paper. *Preprints: International Conference on the Conservation of Library and Archive Materials and the Graphic Arts*, Cambridge, 1980.

Davis, E.A. *Fiber Arts Market and Fiber Artists: As Viewed by Interior Designers and Architects of Five Major Cities in the Southern United States.* Doctoral Dissertation, The University of Tennessee. University Microfilms, Ann Arbor, Mich., 1979.

Drake, G.L. How moisture affects flame retardancy. *Textile Chemist and Colorist* , 1972, 4(12), 43-50.

Drake, G.L., Chance, L.H., Reeves, W.A. A look at flame retardants based on phosphorus compounds. *Journal of Fire and Flammability/ Fire Retardant Chemistry,* 1974, 1, 110-122.

Drake, G.L., Perkins, R.M. and Reeves, W.A. Flame resistant cotton - A status report. *Flammability of Cellulosic Materials, Vol.1.* Hilado, C., Ed. Westport,Conn.: Technomic Publishing Co Inc., 1973.

Drake, G.L. and Reeves, W.A. Flame resistant textiles. *Cellulose and Cellulose Derivatives,* Bikales N.M. and Segal, L., Eds. New York: Wiley Interscience, 1971.

Feller, R.L. Stages in the deterioration of organic materials. *Advances in Chemistry Series, #164.* American Chemical Society, 1977.

Fristrom, R.M. Chemistry, Combustion and Flammability. *Journal of Fire and Flammability,* 1974, 5, 289

Gottlieb, I.M. A theory of flame-retardant finishes. *Textile Research Journal,* 1956, 26, 156-167.

Gordon, B. The fiber of detectives. *Journal of Popular Culture,* 1976, 10(3), 548-559.

Greenwood, C.T. The thermal degradation of starch. *Advances in Carbohydrate Chemistry, Vol.22.* New York: Academic Press, 1967

Hall, A.J. *Textile Finishing.* London: Heywood Books, 1966.

Hendrix, J.E., Drake, G.L. and Reeves, W.A. Effects of fabric weight and construction on Oxygen Index (OI) values for cotton cellulose. *Flammability of Cellulosic Materials,Vol.1.* Hilado, C., Ed. Westport,Conn.: Technomic Publishing Co Inc., 1973.

Hendrix, J.E., Beninate, J.V., Drake, G.L. and Reeves, W.A. Environmental temperatures and Oxygen Index (OI) values for textile fabrics. *Flammability of Cellulosic Materials,Vol.1.* Hilado, C. Ed., Westport,Conn.: Technomic Publishing Co. Inc., 1973.

Hendrix, J.E., Anderson, T.K., Clayton, T.J., Olson, E.S. and Barker, R.H. Flammability measurements and thermal decomposition of textiles. *Flammability of Cellulosic Materials,Vol.1.* Hilado, C. Ed., Westport,Conn.: Technomic Publishing Co. Inc., 1973.

ado, C.J. and Brauer, D.P. The effect of fire retardants on the fire response characteristics of cellulosic materials. *Journal of Fire Retardant Chemistry,* 1978, 5, 196-199.

Hilado, C.J. and Brauer, D.P. The effect of fire retardants on combustability and toxicity. *Journal of Fire Retardant Chemistry,* 1979, 6, 20-25.

Hilado, C.J. and Huttlinger, P.A. Effect of fire retardants on toxicity of off-gases: A review of work done using the NASA-USF method. *Journal of Fire Retardant Chemistry,* 1980, 7, 206-209.

Hilado, C.J. and Huttlinger, P.A. The effect of dyes on the toxicity of gases from burning cotton fabric. *Journal of Combustion Toxicology,* 1982, 9, 97-104.

Hindersinn, R.R. and Witschard, G. The importance of intumescence and char in polymer fire retardance. *Flame Retardancy of Polymeric Materials, Vol.4,* Kuryla, W.C. and Papa, A.J., Eds. New York: Marcel Dekker Inc., 1978.

Holmes, F.H. Flammability and textile products. *The Shirley Link,* England, 1971.

Howe-Echt, K. Questions of style. *Fiberarts,* 1980, 7, 38-43.

Hunnings, R. Fire Retardancy and the Interior Designer. *Fire Retardancy in the Interior Furnishings Industry.* Fire Retardant Chemicals Association, Papers presented at Colonial Williamsburg Lodge, Williamsburg, Virginia., March 14-17, 1976.

Joseph, M.L. *Introductory Textile Science.* New York, Holt, Rinehart and Winston, 1977.

Kasem, M.A. and Rouette, H.K. Flammability and flame retardancy of fabrics: A review. *Flammability of Cellulosic Materials, Vol.1,* Hilado, C.J., Ed. Westport, Conn.: Technomic Publishing Co Inc., 1973.

Kilzer, F.J. Thermal degradation. *Cellulose and Cellulose Derivatives,* Part V., Bikales, N.M. and Segal, L., Eds. New York: Wiley Interscience, 1971.

e, K. and Mohandas, K. Does ammonia act as a flame retardant? *Journal of Fire Sciences,* 1983, 1, 155-157.

Lafontaine, R.H. *Environmental Norms for Canadian Museums, Art Galleries and Archives.* Canadian Conservation Institute, Technical Bulletin #5, Ottawa, 1979.

Lai, Y.Z., Shafizadeh, F., and McIntyre, C.R. Pyrolysis of cellulose after chlorination or addition of zinc chloride. *Cellulose Chemistry and Technology,* Jett, C.A. Ed., American Chemical Society Symposium Series, 48, 1977.

LeBlanc, R.B. Textile flammability update. *Textile Industries,* 1980, 144(2), 82-83.

Leene, J. *Textile Conservation.* London, Butterworths, 1972.

Lewin, M. and Basch, A. Structure, pyrolysis and flammability of cellulose. *Flame-Retardant Polymeric Materials, Vol.2,* Lewin, M., Atlas, S.M. and Pearce, E.M., Eds. New York: Plenum Press, 1978.

Lewin, M. and Sello, S.B. Technology and test methods of flameproofing cellulosics. *Flame-Retardant Polymeric Materials*, Lewin, M., Atlas, S.M. and Pearce, E.M., Eds. New York: Plenum Press, 1975.

Lindsay, J. Cotton goods finshing. *The American Cotton Handbook, Vol.II*, Hamby, D.S., Ed. New York: Interscience Publishers, John Wiley & Sons, 3rd.Ed., 1966.

Little, R.W. *Flameproofing Textile Fabrics.* American Chemical Society Monograph Series 104, New York, Reinhold Publishing Co., 1947.

Little, R.W. Fundamentals of flame retardancy. *Textile Research Journal*, 1951, 21, 901-908.

L , E. Textiles in structures fires: frequency, consequences causes. *American Dyestuff Reporter*, 1981, 70(1), 28-43.

Lyons, J.W. *The Chemistry and Uses of Fire Retardants.* New York: John Wiley & Sons Inc. 1970.

Madorsky, S.L. *Thermal Degradation of Organic Polymers.* Wiley-Interscience, New York. 1964.

Mark, H.F., Atlas, S.M., Shalaby, S.W. and Pearce, E.M. Combustion of polymers and its retardation. *Flame-Retardant Polymeric Materials*, Lewin, M., Atlas, S.M. and Pearce, E.M., Eds. New York: Plenum Press, 1975.

Marsh, J.T. *An Introduction to Textile Finishing.* London: Chapman and Hall Ltd., 1957.

Mayer, F. Flame retardant finishing. *CIBA-Geigy Review*, 1974, 3, 38-40.

McCullough, E.A. *Flammability Characteristics of Layered Fabric Assemblies.* Doctoral dissertation, University of Tennessee, 1978. University Microfilms, Ann Arbor, Michigan.

McKinnon, G.P. Ed. *Fire Protection Handbook, 14th Edit.* Boston, Mass.: National Fire Protection Association, 1976.

Miles, T.D. and Delasanta, A.C. Durable non-reactive FR finishes for cotton. *Textile Research Journal*, 1968, 38(3), 273

Miller, B. and Martin, J.R. Ignition of polymers. *FlameRetardant Polymeric Materials*, Lewin, M., Atlas, S.M. and Pearce, E.M. Eds., New York, Plenum Press, 1978.

Miller, B., Martin, J., Goswani, B. and Meiser, C. Effects of moisture on flammability characteristics of textile materials. *Textile Research Journal*, 1975, 4, 328-337.

National Research Council of Canada. *National Fire Code of Canada.* NRCC, Ottawa, 1977.

Organization for Economic Cooperation and Development. *Safety Requirements Concerning the Flammability of Textile Products.* OECD, Paris, France, 1977.

Ove, J.A. *Designing an Original Large Textile Hanging for a Specific Public Building.* Masters thesis, Iowa State University, Ames, Iowa, 1978.

Parkin, J. *Art in Architecture:Art for the Built Environment in the Province of Ontario.* Boyle, W.J.S., Ed. Visual Arts Ontario, 1982.

Peters, R.H. *Textile Chemistry, Vol.1, The Chemistry of Fibres.* New York: Elsevier, 1963.

Peters, R.H. and Still, R.H. Some aspects of the degradation behavior of polymers used in textile applications. *Applied Fibre Science, Vol.2.* Happey, F., Ed. London: Academic Press Inc., 1979.

Pintauro, E.A. *Ignition Processes in Single and Multicomponent Textile Structures.* Masters thesis, Cornell University, 1978.

Pitts, J.J. Inorganic flame retardants and their mode of action. in *Flame Retardancy of Polymeric Materials, Vol.1.,* Kuryla, W. and Papa, A., Eds. New York: Marcel Dekker Inc., 1973.

Platus, L. Flameproofing fibers. *Shuttle, Spindle and Dyepot,* 1977, VIII(2), 29-30.

Pomerantz, L. *Is Your Contemporary Painting More Temporary Than You Think.* Chicago, Ill.: International Book Co., 1962.

Rebenfeld, L., Miller, B. and Martin, J.R. The thermal and flammability behavior of textile materials. *Applied Fibre Science, Vol.2,* Happey, F., Ed. London: Academic Press Inc., 1979.

Reeves, W.A. Durable phosphorus-containing flame retardants for cellulosic textiles. *Textile Chemist and Colorist,*1972, 4(2), 45-49.

Reeves, W.A. What next for fire resistant textiles? *American Dyestuff Reporter,* 1983, 72(1), 16-17.

Reeves, W.A. and Drake, G.L. *Flame Resistant Cotton.* Merrow Monographs, Textile Technology Series, Watford, England: Merrow Publishing Co Ltd., 1971.

Reeves, W.A. and Guthrie, J.D. Chemical modification. *The American Cotton Handbook, Vol.II,* Hamby, D.S.,Ed. New York: Interscience Publishers, John Wiley & Sons, 3rd.Ed., 1966.

Robinson, H.M. and Reeves, W.A. A survey of the effect of light on cotton and other cellulosic fabrics. *American Dyestuff Reporter*, 1961, 50, 17-31.

Shafizadeh, F. Pyrolysis and combustion of cellulosic materials. *Advances in Carbohydrate Chemistry, Vol.23.* New York: Academic Press, 1968.

Shafizadeh, F., Chin, Ping-Sen and DeGroot, W.F. Mechanistic evaluation of flame retardants. *Journal of Fire and Flammability/ Fire Retardant Chemistry*, 1975, 2, 195-202.

Shenouda, S.G. The structure of cotton cellulose. *Applied Fibre Science, Vol.3.*, Happey, F., Ed. London: Academic Press, 1979.

Soignet, D.M. and Benerito, R.R. Surface and bulk properties of FR cotton fabrics, Part II: Effect of exposure to atmospheric conditions. *Journal of Fire Retardant Chemistry*, 1979, 6, 225-238. SPSSx. *User's Guide.* SPSS Inc., Chicago, Ill., 1983.

Solomons, T.W.G. *Organic Chemistry.* New York: John Wiley & Sons Inc., 1976.

TAPPI. *Standards and Suggested Methods.* Technical Association of the Pulp and Paper Industry, New York. 1968

Tesoro, G. Chemical technology in the "Tris" experience. *Journal of Fire Retardant Chemistry*, 1979, 6, 239-248.

Timpa, J.L., Segal, L. and Drake, G.L. Dyes and flame-retardant cotton textiles. *Journal of Fire and Flammability/Fire Retardant Chemistry*, 1975, 2, 83-93.

Tovey, H. and Katz, R. Textile products in structures fires: frequency, consequences and causes. *Textile Chemist and Colorist*, 1981, 13(5), 26.

Tuve, R.L. *Principles of Fire Protection Chemistry.* Boston, Mass., National Fire Protection Association, 1976.

Uehara, Y. and Yanai, E. Smoke, carbon monoxide and carbon dioxide from cellulosic materials treated with DAP in a fire environment. *Flammability of Cellulosic Materials, Vol.1*, Hilado, C., Ed. Westport Conn.: Technomic Publishing Co Inc., 1973.

Vail, S.L., Daigle, D.J. and Frank, A.W. Chemistry of hydroxymethyl phosphorus compounds, part I. *Textile Research Journal*, 1982, 52(11), 671-677.

Valko, E.I. Penetration of fabrics. *Chemical Aftertreatment of Textiles*, Mark, H., Wooding, N.S. and Atlas, S.M., Eds. New York: Wiley Interscience, John Wiley & Sons Inc., 1971.

Vigo, T.L. Preservation of natural textile fibers - historical perspectives. *Advances in Chemistry Series, #164*, American Chemical Society, 1977.

Warner, R.K. Testing of physical and performance properties of yarns and fabrics. *The American Cotton Handbook, Vol.II,* Hamby, D.S. Ed. New York: Interscience Publishers, John Wiley & Sons, 3rd.Edn., 1966.

Weaver, J.W. The burning behavior of marginal flame retardant fabrics. *Textile Chemist and Colorist,* 1972, 4(5), 116-118.

Welker, J.R. The pyrolysis and ignition of cellulosic materials : A literature review. *Flammability of Cellulosic Materials, Vol.1.* Hilado, C., Ed. Westport, Conn.: Technomic Publishing Co Inc. [illegible]3.

Williams, A. *Flame Resistant Fabrics.* Noyes Data Corporation, New Jersey, 1974.

Windholz, M., Ed. *The Merck Index, Ninth Edition.* Rahway, New Jersey: Merck & Co Inc., 1976.

Yeager, C.C. and Chapin, J.C. A systems approach to flame retardacy. *Textile Chemist and Colorist,* 1972, 4(4), 39-43.

REFERENCE NOTES

1. Kimura, M. *Artists' Attitudes Towards Art Preservation.* Interim Report, Alberta Culture, Visual Arts, March, 1984.

2. Thomas, J. *Commissioning Textile Art Works.* Alberta Culture, Visual Arts. Feb. 1980.

3. Clark, K. and Dillon, P. *Preliminary Investigation of a Large Modern Wall Hanging.* Paper presented at the Harper's Ferry Textile Preservation Conference, 1981.

4. Suzuki, A. Personal Communication, Oct.18th, 1983.

5. Thomas, J. *Commissioned Textile Works: A follow up report.* Alberta Culture, Visual Arts. June, 1980.

6. Watson, S. Personal communication, Apr.11th, 1983.

APPENDIX I

Chemical Analyses of Proprietary Fire Retardant Compounds

CHEMICAL AND MATERIALS ENGINEERING DEPARTMENT
CHEMISTRY PRODUCT EVALUATION LAB REPORT

LAB SAMPLE NO.: 700-831.C through 702-831.C.

Reference: V. Osinchuk, I.D.D. for: University of Alta Household Economics.
(Company & address)

Submitted by: Same

Date Submitted: Nov. 15/83

700 831.C	V-15-11-105	Flamegard DSH
701-831.C	V-15-11-106	STS (not used in this study)
702-831.C	V-15-11-107	X-12

1. Spot Tests.

Sample No:	Test results and Observations			
	Chloride	Carbonate	Sulfate	Phosphate
700-831.C. (Flamegard DSH)	positive	negative	positive	positive
701-831.C. (STS)	positive	negative	positive	negative

Note: 1. 700-831.C. & 701-831.C contain a lot of chloride
2. 701-831.C contains more sulfate than 700 831.C
3. tested for PO_4 using ammonium molybdate. 700 831.C turned yellow · posi

2. Emission Spec Results.

	Sodium
700 831.C	positive
701-831.C	positive

Completed Date: Dec. 12/83

Signed: K. Brinchly

CHEMICAL AND MATERIALS ENGINEERING DEPARTMENT

CHEMISTRY PRODUCT EVALUATION LAB REPORT

LAB SAMPLE NO.: 700-831.C through 702-831.C

Reference:
(Company & address)

Submitted by: ______________________ Date Submitted: ______________

3. XRF results

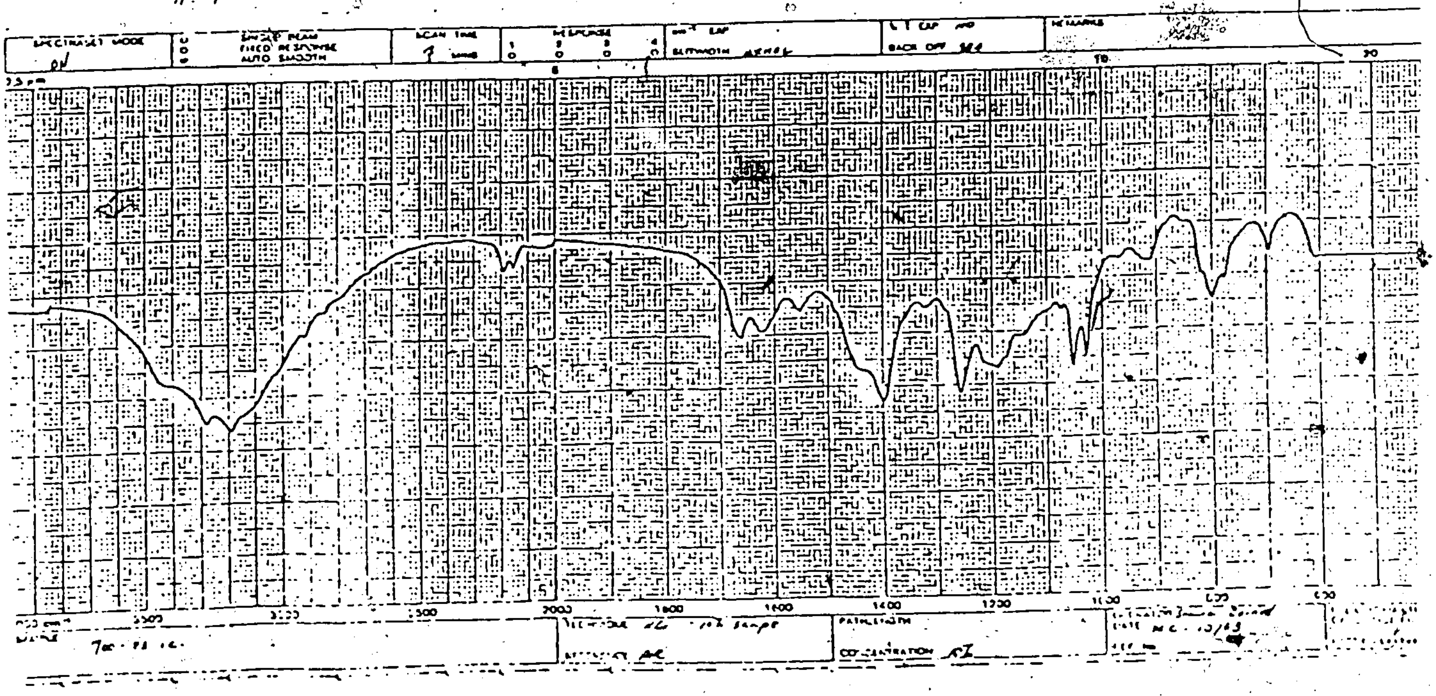
	Major	Minor	Trace
700-831.C. [illegible]	S, Br	Ca	—
701-831 C srs	P	S	Ca
702-831.C x-12	S	P	Ca, Fe

4 I.C.P. results

	Boron ug/ml	Na
700-831.C	11,200 Boron	9000 ug/
701-831.C	<01	77

5 I.R.'s:

a. 100·831.C



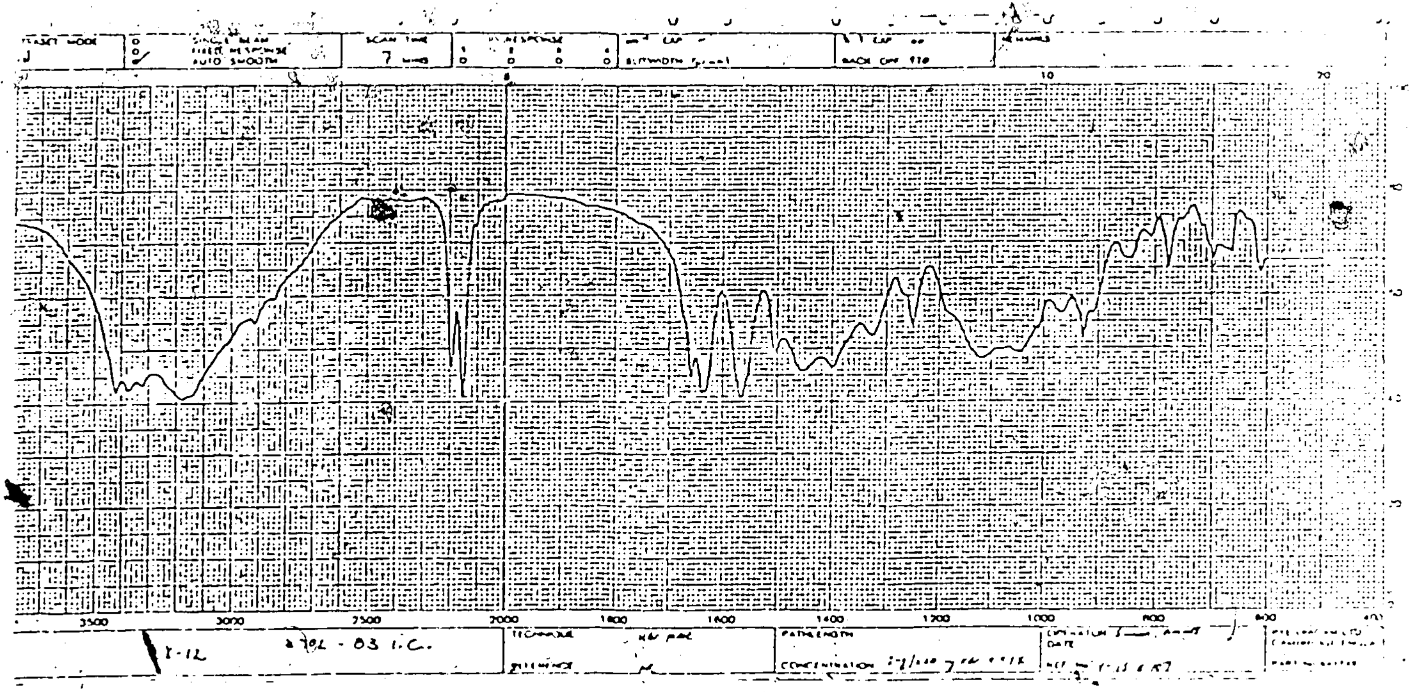
CHEMICAL AND MATERIALS ENGINEERING DEPARTMENT
CHEMISTRY PRODUCT EVALUATION LAB REPORT

LAB SAMPLE NO.: 100-831C through 102-831C

Reference:
(Company & address)

Submitted by: ______________________ Date Submitted: ______________

C. 102-831.C V-15-11-107 X-12



Completed Date: ______________________ Signed: ______________

APPENDIX II

Dyeing Method used for Procion® Fiber Reactive Dyes

Long Method for Dyeing

PROCION M-SERIES DYES ONLY!

EQUIPMENT

PLASTIC, STAINLESS STEEL, or ENAMEL DYE CONTAINER: plastic dishpans, wastebaskets, or garbage cans work especially well. Must be large enough to comfortably hold the piece being dyed.
SCALE or MEASURING SPOONS
MIXING CUPS
RUBBER GLOVES

SUPPLIES

CALGON® (only if water is hard; use 1 Tbsp. to 1 gal. water)
PROCION M-SERIES DYE
TABLE SALT
SODA ASH (SAL SODA or pure WASHING SODA) NOT BAKING SODA!

NOTE! Do not use Arm & Hammer brand "Super Washing Soda" sold in super markets! Recently the manufacturer began to include bleach and other chemicals which prevent the dyes from fixing properly. Use only pure soda ash or washing soda!

PREPARATION

1. Except for fabric which has been especially prepared for dyeing (P.F.P. goods), all fabric should be washed in hot water and detergent before dyeing to remove all sizing and starch. The fabric should be rinsed well, but need not be dry. (If shrinkage is critical, even P.F.P. fabrics should be washed.)

2. Cellulose fibres, particularly viscose rayon and mercerized cotton, dye significantly better than other types of fibres. (Mercerizing is a special treatment in which the cotton fabric is run through a series of lye baths while being held under tension. This results in a change in the structure of the cellulose in the cotton which dramatically increases the affinity of the fibre for dye.) Unbleached and unmercerized cottons like muslin will not dye nearly as deeply as bleached and mercerized fabrics. Also, with PROCION® M-SERIES DYES, silk will only dye pastel to medium shades of color using immersion techniques. To achieve deep colors on silk, try direct application techniques, i.e., printing or painting, instead. (The PROCION® M-SERIES DYES are especially well suited for this purpose!)

3. Proportions of dye and salt in the dyeing recipe are based on one pound (450 grams) of dry fabric, approximately 2-4 yards, 36" wide.

4. This "long" method for dyeing is more economical in terms of the amount of dye required, i.e., deeper, more intense shades can be obtained with smaller amounts of dye. Remember, too, the the depth of shade is controlled by the amount of dye powder used, NOT the amount of time in the dyebath. Removing the fabric from the dye bath before the dye process is complete in an attempt to get lighter shades will only result in loss of washfastness.

5. Note that each color varies slightly in its nature. For instance, PROCION® M-SERIES colors P117 NAVY BLUE MX-2GA, P115 BROWN MX-CRA, P113 BLACK MX-CWA, P114 TURQUOISE MX-G and P120 RUST MX-GRN tend to be "weaker" colors and for really deep shades it may be necessary to use more dye and salt than called for in the recipe. Up to a certain point, the more salt you add, the deeper the shade you are going to get. (Check the table on the top of the next page.) Salt is relatively cheap. Don't skimp if you want dark shades.

PROCION® M-SERIES DYES P104 FUCHSIA MX-8B, P122 BRILLIANT YELLOW MX-8G and P123 CERULEAN BLUE MX-G CONC are more intense than other colors and require less dye and salt to achieve full shades of color.

PROCEDURE for 1 pound (450 grams) of dry fabric.

SHADE	AMT OF DYE	%owf	AMOUNT OF SALT	%owf
PALE	3/4 tsp (2.25 g)	.5%	3 Tbsp (90 g)	20%
MEDIUM	2¼ tsp (6.75 g)	1.5%	6-9 Tbsp (180-270 g)	40-60%
DARK	6 tsp (18 g)	4 %	1-1½ cups (480-720 g)	106-160%
REALLY DEEP	12+ tsp (36+ g)	8+ %	2½+ cups (1200+ g)	266%

NOTE: %owf = percent of weight of fibre

1) Place the required amount of dye for the shade desired into a one-quart (1 litre) Pyrex measuring cup, add a small amount of cold water and work into a smooth paste free from lumps with a stainless steel or plastic spoon.

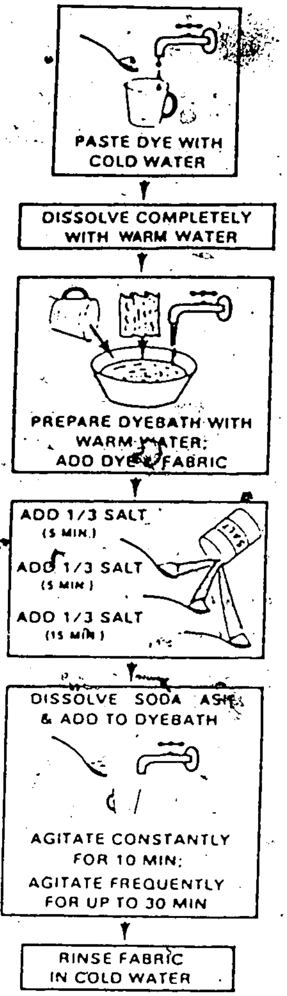
2) Add enough water at a temperature of 40°C (105°F, slightly warmer than body temperature) to dissolve the dye completely. This will require at least ½-cup (125 ml) of water for each teaspoon (3 grams) of dye.

3) For each pound (450 g.) of fabric use 2½ gallons (9 litres) of water at 40°C (105°F). If the water in your area is "hard", add Calgon water softener at this point. Add the dissolved dye and stir. Then add the cleaned fabric and stir frequently for 10 minutes.

4) Add the required amount of SALT to the dyebath in 3 equal parts with 5 minutes between additions. Continue to stir frequently for 15 minutes after all the SALT is in. You should notice the fabric becoming darker with each addition as the salt forces the fabric to absorb the dye. At this point, if the fabric does not appear dark enough, additional salt and/or dye can be added. Be sure to pre-dissolve the salt or dye in warm water before adding. Wait 10-15 minutes before judging the color again. Also remember that wet fabric looks 2-3 shades darker than dry fabric.

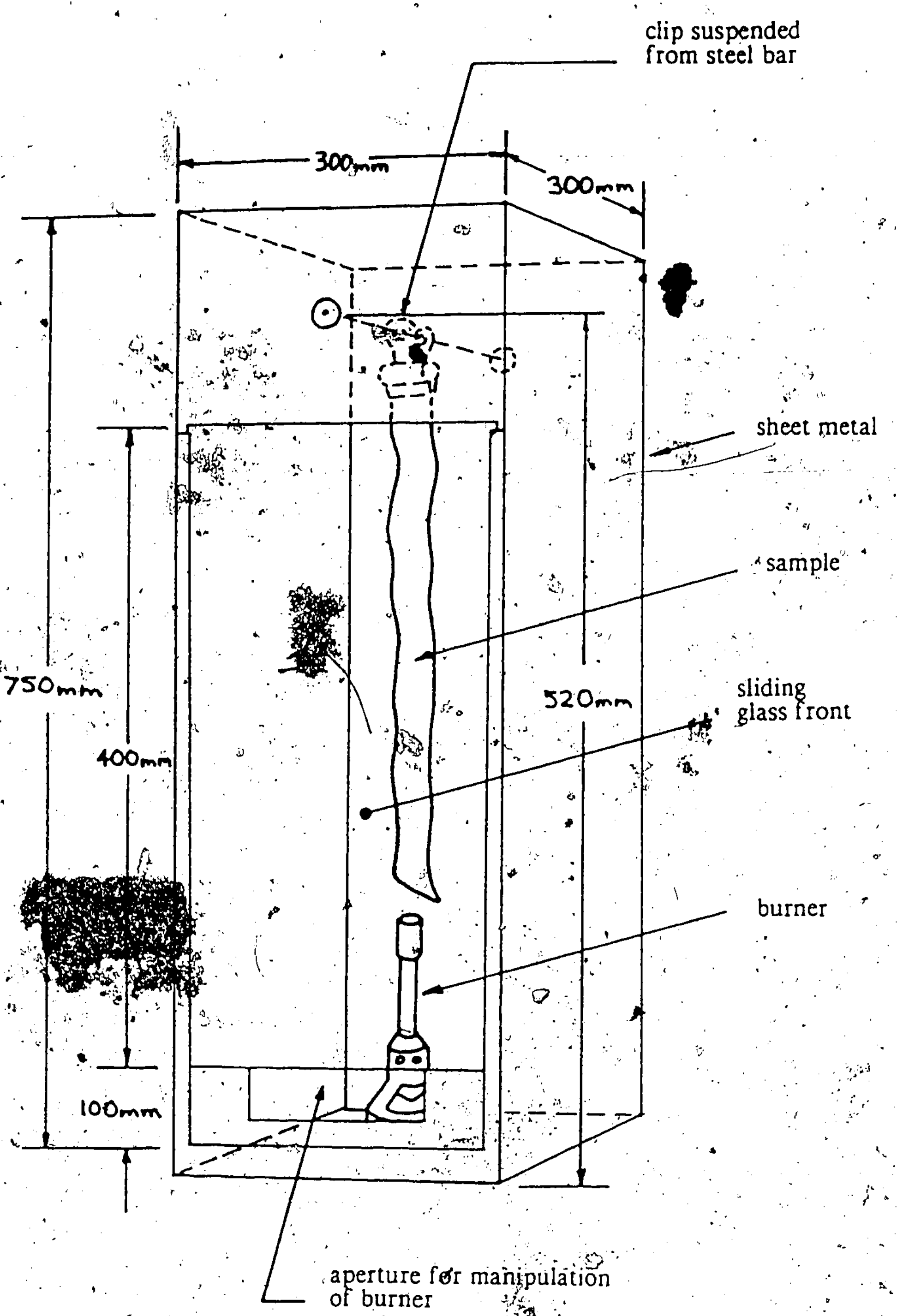
5) For each pound (450 g.) of fabric, dissolve 3 tablespoons (40 g.) of SODA ASH in a small amount of warm water and add to the dyebath. The addition of the SODA ASH starts the dye reaction and since most of the reaction takes place in the first 10 minutes after the SODA ASH is added, it is important to be agitating or stirring during this period to prevent streaking or blotching. Continue to stir every 5 minutes or so for at least 30 minutes, and up to 1 hour to achieve maximum depth of shade.

6) Rinse the dyed cloth well in cold running water



APPENDIX III

Apparatus used in the Vertical Flame Test



Apparatus used in the vertical flame test

APPENDIX IV

Statistical Analyses

Table IV.1 Analysis of variance for dimensional change(cm) in the warp direction

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	3	.1646	.0549	21.2366	.0000
WITHIN GROUPS	20	.0517	.0026		
TOTAL	23	.2162			

Table IV.2 Duncan's Multiple Range Test for dimensional change(cm) in the warp direction

```
  (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                              w B X D
                              a / - S
                              t B 1 H
                              e A 2
                              r /
                                D
                                A
    MEAN       GROUP            P

    -.3500     water
    -.2833     B/BA/DAP
    -.1667     X-12           * *
    -.1500     DSH            * *

  HOMOGENEOUS SUBSETS    (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                          DO NOT DIFFER BY MORE THAN THE SHORTEST
                          SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET   1

GROUP         water          B/BA/DAP
MEAN        -0.3500         -0.2833
- - - - - - - - - - - - - - - - - - -
SUBSET   2

GROUP         X-12           DSH
MEAN        -0.1667         -0.1500
- - - - - - - - - - - - - - - - - - -
```

Table IV.3 Analysis of variance for dimensional change(cm) in the weft direction

```
                                  SUM OF        MEAN           F       F
        SOURCE          D.F.     SQUARES       SOUARES       RATIO   PROB.

BETWEEN GROUPS            3        .2050         .0683       7.8846   .0012

WITHIN GROUPS            20        .1733         .0087

TOTAL                    23        .3783
```

Table IV.4 Duncan's Multiple Range Test for dimensional change(cm) in the weft direction

```
  (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                          w B X D
                          a / - S
                          t B 1 H
                          e A 2
                          r /
                            D
                            A
      MEAN      GROUP       P

      -.2000    water
      -.1667    B/BA/DAP
       .0000    X-12      * *
       .0000    DSH       * *

  HOMOGENEOUS SUBSETS    (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                          DO NOT DIFFER BY MORE THAN THE SHORTEST
                          SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET  1

GROUP        water         B/BA/DAP
MEAN        -0.2000       -0.1667
- - - - - - - - - - - - - - - - -

SUBSET  2

GROUP       X-12          DSH
MEAN        0.0           0.0
- - - - - - - - - - - - - - - - -
```

Table IV.5 Analysis of variance for tensile strength before aging

```
                                         SUM OF         MEAN              F       F
        SOURCE                D.F.      SQUARES        SQUARES          RATIO   PROB.

BETWEEN GROUPS                  3       863.9610       287.9870       10.6097   .0000

WITHIN GROUPS                  36       977.1740        27.1437

TOTAL                          39      1841.1350
```

Table IV.6 Duncan's Multiple Range Test for tensile strength before aging

```
  (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                                  B w X D
                                  / a - S
                                  B t 1 H
                                  A e 2
                                  / r
                                  D
                                  A
     MEAN       GROUP             P

   153.7300     B/BA/DAP
   156.3700     water
   159.4200     X-12
   166.1800     DSH               * * *

 HOMOGENEOUS SUBSETS    (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                         DO NOT DIFFER BY MORE THAN THE SHORTEST
                         SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET  1

GROUP        B/BA/DAP      water         X-12
MEAN       153.7300     156.3700      159.4200
- - - - - - - - - - - - - - - - - - - - - - - -

SUBSET  2

GROUP        DSH
MEAN       166.1800
- - - - - - - - - - -
```

Table IV.7 Analysis of variance for tensile strength after aging

```
                                    SUM OF        MEAN             F        F
         SOURCE              D.F.   SQUARES       SQUARES          RATIO    PROB.

BETWEEN GROUPS                 3    45954.5940    15318.1980   1865.4063   .0000

WITHIN GROUPS                 36      295.6220        8.2117

TOTAL                         39    46250.2160
```

Table IV.8 Duncan's Multiple Range Test for tensile strength after aging

```
  (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                               X D B w
                               - S / a
                               1 H B t
                               2   A e
                                   / r
                                   D
                                   A
    MEAN        GROUP              P

   22.5600      X-12
   23.3400      DSH
   79.3500      B/BA/DAP       * *
   99.1900      water          * * *

 HOMOGENEOUS SUBSETS     (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                          DO NOT DIFFER BY MORE THAN THE SHORTEST
                          SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET  1

GROUP          X-12          DSH
MEAN         22.5600       23.3400
- - - - - - - - - - - - - - - - -

SUBSET  2

GROUP          B/BA/DAP
MEAN         79.3500
- - - - - - - - - - -

SUBSET  3

GROUP          water
MEAN         99.1900
- - - - - - - - - - -
```

Table IV.9 Analysis of variance for pH before aging

```
                                     SUM OF        MEAN              F        F
        SOURCE            D.F.      SQUARES       SQUARES          RATIO    PROB.

BETWEEN GROUPS              3       14.4811        4.8270       375.4352   .0000

WITHIN GROUPS              24         .3086         .0129

TOTAL                      27       14.7896
```

Table IV.10 Duncan's Multiple Range Test for pH before aging

```
  (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                                 D X w B
                                 S - a /
                                 H 1 t B
                                   2 e A
                                     r /
                                       D
                                       A
     MEAN        GROUP                 P

     5.5429      DSH
     6.3429      X-12            *
     7.2286      water           * *
     7.3000      B/BA/DAP        * *

  HOMOGENEOUS SUBSETS    (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                          DO NOT DIFFER BY MORE THAN THE SHORTEST
                          SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)


SUBSET  1

GROUP         DSH
MEAN          5.5429
- - - - - - - - - - -

SUBSET  2

GROUP         X-12
MEAN          6.3429
- - - - - - - - - - -

SUBSET  3

GROUP         water          B/BA/DAP
MEAN          7.2286         7.3000
- - - - - - - - - - - - - - - - - -
```

Table IV.11 Analysis of variance for pH after aging

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	3	9.9738	3.3246	2659.6667	.0000
WITHIN GROUPS	4	.0050	.0012		
TOTAL	7	9.9788			

Table IV.12 Duncan's Multiple Range Test for pH after aging

```
  (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                              X D B w
                              - S / a
                              1 H B t
                              2   A e
                                  / r
                                  D
                                  A
      MEAN       GROUP            P

      3.8000     X-12
      4.0500     DSH          *
      6.1000     B/BA/DAP     * *
      6.2000     water        * *

  HOMOGENEOUS SUBSETS   (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                         DO NOT DIFFER BY MORE THAN THE SHORTEST
                         SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET  1

GROUP        X-12
MEAN         3.8000
- - - - - - - - - -

SUBSET  2

GROUP        DSH
MEAN         4.0500
- - - - - - - - - -

SUBSET  3

GROUP        B/BA/DAP      water
MEAN         6.1000        6.2000
- - - - - - - - - - - - - - - - -
```

Table IV.13 Analysis of variance for colour change of undyed cotton

```
                                     SUM OF        MEAN              F        F
       SOURCE              D.F      SQUARES      SQUARES          RATIO    PROB.

BETWEEN GROUPS               7    1450.1177     207.1597        77.0923    .0000

WITHIN GROUPS               45     120.9224       2.6872

TOTAL                       52    1571.0401
```

Table IV.14 Duncan's Multiple Range Test for colour change of undyed cotton

```
 (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                             w D X B B w X D
                             a S - / / a - S
                             t H 1 B B t 1 H
                             e   2 A A e 2
                             r u   / / r   a
                                 u D D   a
                             u           a
    MEAN        GROUP                u a

    .0000       water u
    .5960       DSH u
    .9120       X-12 u
    .9900       B/BA/D u
   2.2850       B/BA/D a
   2.5220       water a
  10.5450       X-12 a       * * * * * *
  13.1960       DSH a        * * * * * * *
```

u = unaged

a = aged

Table IV.15 Analysis of variance for colour change of cotton dyed red

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	3	30.6488	10.2163	516.4291	.0000
WITHIN GROUPS	16	.3165	.0198		
TOTAL	19	30.9653			

Table IV.16 Duncan's Multiple Range Test for colour change of cotton dyed red

```
  (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                           w D X B
                           a S - /
                           t H 1 B
                           e   2 A
                           r     /
                                 D
                                 A
     MEAN       GROUP            P

     .0000      water
    2.6960      DSH        *
    2.8180      X-12       *
    3.0240      B/BA/DAP   * *

  HOMOGENEOUS SUBSETS     (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                           DO NOT DIFFER BY MORE THAN THE SHORTEST
                           SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET   1

GROUP         water
MEAN          0.0
- - - - - - - - - -

SUBSET   2

GROUP         DSH             X-12
MEAN          2.6960          2.8180
- - - - - - - - - - - - - - - - - -

SUBSET   3

GROUP         X-12            B/BA/DAP
MEAN          2.8180          3.0240
- - - - - - - - - - - - - - - - - -
```

Table IV.17 Analysis of variance for colour change of cotton dyed yellow

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	3	19.2955	6.4318	95.1806	.0000
WITHIN GROUPS	16	1.0812	.0676		
TOTAL	19	20.3767			

Table IV.18 Duncan's Multiple Range Test for colour change of cotton dyed yellow

```
   (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                             w B X D
                             a / - S
                             t B 1 H
                             e A 2
                             r /
                               D
                               A
     MEAN       GROUP          P

     .0000      water
     .7440      B/BA/DAP     *
    1.7180      X-12         * *
    2.6000      DSH          * * *

  HOMOGENEOUS SUBSETS    (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                          DO NOT DIFFER BY MORE THAN THE SHORTEST
                          SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET   1

GROUP         water
MEAN          0.0
- - - - - - - - - - - -

SUBSET   2

GROUP         B/BA/DAP
MEAN          0.7440
- - - - - - - - - - - -

SUBSET   3

GROUP         X-12
MEAN          1.7180
- - - - - - - - - - - -

SUBSET   4

GROUP         DSH
MEAN          2.6000
- - - - - - - - - - - -
```

Table IV.19 Analysis of variance for colour change of cotton dyed blue

```
                                   SUM OF          MEAN               F        F
        SOURCE          D.F.       SQUARES        SQUARES           RATIO    PROB.

BETWEEN GROUPS            3        25.0702         8.3567        259.0028    .0000

WITHIN GROUPS            16          .5162          .0323

TOTAL                    19        25.5864
```

Table IV.20 Duncan's Multiple Range Test for colour change of cotton dyed blue

```
  (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                            w X D B
                            a - S /
                            t 1 H B
                            e 2   A
                            r     /
                                  D
                                  A
   MEAN        GROUP              P

    .0000      water
   2.2600      X-12         *
   2.3260      DSH          *
   2.9460      B/BA/DAP     * * *

 HOMOGENEOUS SUBSETS    (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                         DO NOT DIFFER BY MORE THAN THE SHORTEST
                         SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET  1

GROUP        water
MEAN         0.0
- - - - - - - - - - -

SUBSET  2

GROUP        X-12           DSH
MEAN         2.2600         2.3260
- - - - - - - - - - - - - - - - - - -

SUBSET  3

GROUP        B/BA/DAP
MEAN         2.9460
- - - - - - - - - - -
```

Table IV.21 Analysis of variance for colour change of cotton dyed green

```
                                       SUM OF          MEAN              F        F
        SOURCE                D.F.     SQUARES         SQUARES           RATIO    PROB.

BETWEEN GROUPS                  3       10.9236          3.6412      148.0009    .0000

WITHIN GROUPS                  16         .3936           .0246

TOTAL                          19       11.3172
```

Table IV.22 Duncan's Multiple Range Test for colour change of cotton dyed green

```
    (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                               w B X D
                               a / - S
                               t B 1 H
                               e A 2
                               r /
                                 D
                                 A
     MEAN        GROUP           P

      .0000      water
      .9160      B/BA/DAP      *
     1.2800      X-12          * *
     2.0560      DSH           * * *

  HOMOGENEOUS SUBSETS    (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                          DO NOT DIFFER BY MORE THAN THE SHORTEST
                          SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)


SUBSET  1

GROUP        water
MEAN         0.0
- - - - - - - - - - -

SUBSET  2

GROUP        B/BA/DAP
MEAN         0.9160
- - - - - - - - - - -

SUBSET  3

GROUP        X-12
MEAN         1.2800
- - - - - - - - - - -

SUBSET  4

GROUP        DSH
MEAN         2.0560
- - - - - - - - - - -
```

Table IV.23 Analysis of variance for colour change of cotton dyed orange

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	3	28.4383	9.4794	263.5555	.0000
WITHIN GROUPS	16	.5755	.0360		
TOTAL	19	29.0138			

Table IV.24 Duncan's Multiple Range Test for colour change of cotton dyed orange

```
   (*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                           w X D B
                           a - S /
                           t 1 H B
                           e 2   A
                           r     /
                                 D
                                 A
      MEAN      GROUP            P

       .0000    water
      1.4380    X-12       *
      2.4580    DSH        * *
      3.1740    B/BA/DAP   * * *

  HOMOGENEOUS SUBSETS   (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                         DO NOT DIFFER BY MORE THAN THE SHORTEST
                         SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET  1

GROUP        water
MEAN         0.0
- - - - - - - - - -

SUBSET  2

GROUP        X-12
MEAN         1.4380
- - - - - - - - - -

SUBSET  3

GROUP        DSH
MEAN         2.4580
- - - - - - - - - -

SUBSET  4

GROUP        B/BA/DAP
MEAN         3.1740
- - - - - - - - - -
```

Table IV.25 Analysis of variance for colour change of cotton dyed purple

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	3	10.9727	3.6576	70.0312	.0000
WITHIN GROUPS	16	.8356	.0522		
TOTAL	19	11.8083			

Table IV.26 Duncan's Multiple Range Test for colour change of cotton dyed purple

```
(*) DENOTES PAIRS OF GROUPS SIGNIFICANTLY DIFFERENT AT THE 0.010 LEVEL

                                 w D B X
                                 a S / -
                                 t H B 1
                                 e   A 2
                                 r   /
                                     D
                                     A
    MEAN         GROUP               P

     .0000       water
    1.3900       DSH             *
    1.5260       B/BA/DAP        *
    1.9840       X-12            * * *

 HOMOGENEOUS SUBSETS      (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS
                           DO NOT DIFFER BY MORE THAN THE SHORTEST
                           SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET  1

GROUP         water
MEAN          0.0
- - - - - - - - - - -

SUBSET  2

GROUP         DSH            B/BA/DAP
MEAN          1.3900         1.5260
- - - - - - - - - - - - - - - - - - - -

SUBSET  3

GROUP         X-12
MEAN          1.9840
- - - - - - - - - - -
```