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Chastity Danielle Newsome, Student Dr. Elizabeth P. Easter, Major Professor Name not available, Director of Graduate Studies

ABSTRACT OF THESIS

EVALUATION OF MOISTURE BARRIERS FOR FIRE FIGHTING TURNOUT GEAR ASSESSMENT OF PRODUCT FAILURE AND TEST METHOD DEVELOPMENT PREDICTING FAILURE MODES

The purpose of this study was to investigate the failures seen in the moisture barrier of fire fighting turnout gear. Moisture barriers taken from garments in field were evaluated to establish a baseline for comparison. Moisture barriers were exposed as part of a three-piece ensemble and as a single layer moisture barrier to instrumental light exposure in a Carbon Arc Fade-ometer and natural sunlight exposure according to AATCC and ASTM test methods. After exposure, moisture barriers were visually examined using stereo and compound microscopes. A performance measurement was conducted on the exposed moisture barriers using a modified NFPA Hydrostatic Water Penetration Resistance Test. Results of the instrumental and natural sunlight exposures were compared to the failed garments from the field.

The results showed that moisture barriers were degraded by ultraviolet light and replicated some of the results seen in the field to predict failures. Based on the results of this study, suggestions were made for future research for developing a test method for predicting moisture barrier failures.

EVALUATION OF MOISTURE BARRIERS FOR FIRE FIGHTING TURNOUT GEAR ASSESSMENT OF PRODUCT FAILURE AND TEST METHOD DEVELOPMENT PREDICTING FAILURE MODES

By

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Date

THESIS

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The Graduate School

University of Kentucky

EVALUATION OF MOISTURE BARRIERS FOR FIRE FIGHTING TURNOUT GEAR ASSESSMENT OF PRODUCT FAILURE AND TEST METHOD DEVELOPMENT PREDICTING FAILURE MODES

THESIS

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Interior Design, Merchandizing and Textiles at the University of Kentucky

By

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Chapter One

Introduction

Flames are raging, temperatures are as high as 1000° F, smoke is so thick flames can only be seen as an orange glow, and the fire fighter must try and locate the seat of the fire to apply water for extinguishing the fire. This type of extreme condition gives rise to the need for protective clothing for fire fighters. Since they never know what to expect until they arrive on the scene, their clothing must protect them no matter how severe the conditions. The protective gear used by fire fighters consists of different items that aid in their protection, including helmets, gloves, boots, and turnout gear consisting of a coat and pant or coveralls. Fire is not the only extreme condition faced by fire fighters and their gear. Fire fighters work year-round, therefore they are exposed to heat, cold, and other weather conditions. However, fire fighters don't have seasonal gear. Protective clothing worn by fire fighters keeps them warm, prevents them from getting wet and provides them minimum protection from burns by flame, scald burns, and injuries from sharp and falling objects.

The National Fire Protection Association (NFPA) reported that 87,500 fire fighters were injured in 1999. Of those injuries 4,865 were burn related; 4,420 were due to thermal stress, which includes heat exhaustion and frostbite; and the other 78,215 were smoke inhalation, other respiratory distress, eye irritation, wounds, fractures, heart attack or stroke, muscular pain and others. These fire fighter injuries reflect a 1.1% increase over 1998. The increase in injuries could be a result of many factors, such as the fire fighter not being aware of the dangerous temperatures, prolonged and direct exposure to heat and flames and the limitations of their turnout gear (Karter and Badger, 2000).

In 1975 the National Fire Protection Association developed the first edition of the NFPA 1971 standard for fire fighting protective clothing. The NFPA publishes consensus standards for certification, inspecting, testing, labeling, and design requirements. These standards also specify some requirements for flame and heat resistance, water absorption resistance, tear and tensile strength. The requirements set forth in the NFPA 1971 standard must be met or exceeded by fire protective clothing manufacturers and by fire departments, which add equipment or clothing to the ensemble.

Although the NFPA process has designed these standards for the industry, protective clothing will not meet the requirements of these standards for the lifetime of the fire fighter gear. Wear and tear of protective gear occurs when exposed to the different elements of fire fighting and normal everyday wear, including washing. Since the protective properties of turnout gear cannot be maintained forever, it is important to know when one's gear is no longer offering sufficient protection and must be replaced.

Justification

Burn injuries, which occur during fire fighting activities, stem from several factors: thermal exposure; movement and actions of fire fighters while performing their duties; physiological functions which regulate the building up of heat in the body; and the performance of the protective clothing ensemble (Lawson, 1996). To help prevent burn injuries, turnout gear has gone from canvas and rubber to high tech fibers and microporous materials, such as aramids and polybenzimidazole (PBI), and polytetraflourethylene (PTFE), which are not only flame-resistant, but also more comfortable. With new bench top and thermal mannequin tests, researchers are able to evaluate the materials and the entire protective clothing ensemble under conditions

similar to those which fire fighters face. However, limited procedures are available to evaluate protective clothing materials in use. Because of limited evaluative procedures, little is known about what occurs within the protective gear when exposed to fire fighting environments. Researchers are continually evaluating issues such as moisture transfer and durability in turnout gear as well as heat stress. With more knowledge of what occurs within protective gear, current test methods can be improved and new tests can be developed (Torvi, et al, 1999).

A recent problem experienced by the protective clothing industry was the degradation of Breathe-Tex®, a type of moisture barrier used in fire fighting protective clothing. The problem was recognized by the industry as manufacturers of turnout gear had garments returned from the field that had reportedly failed. The three-layer system was cut and examined to assess the condition of the moisture barriers. The degradation was reportedly apparent within the polyurethane film layer, which was changing color and/or experiencing cracks, flaking or peeling of the film from the substrate. The degradation of Breathe-Tex® was widespread but the cause was not as obvious, because of the pattern of damage seen in garments from the field, that is, garments showed severe damage in areas where light and abrasion were thought to have contributed to the breakdown of the film. In contrast, damage was also apparent in garments that were primarily in storage or had experienced limited actual use but were inside the fire station. For example, pants of the turnout gear had severe damage in the upper sections but virtually none in the inside of legs where no light exposure occurred.

The ramification of this problem is that failure of the moisture barrier layer of turnout gear may cause scalding or burn injuries to fire fighters to occur. Failures in the

moisture barrier prevent the gear from keeping the wearer dry; increasing the potential for scalding, burn injuries, hazardous liquids, and exposure to blood borne pathogens. In some cases where burn injuries have occurred, there may be no sign of damage to the turnout gear (Lawson, 1996). Since the moisture barrier is hidden and protected by the thermal barrier, failures are difficult to detect and fire fighters cannot determine if any degradation due to laundering, high heat exposure, abrasion or light exposure has occurred. Once the degradation begins, the turnout gear may no longer meet National Fire Protection Association (NFPA) minimum performance requirements.

Currently, NFPA Standards specify minimum requirements for the performance of only new protective clothing. How well turnout gear performs after extended use is unknown. The only study conducted on used protective clothing is that of Vogelpohl which found used garments, which had been in use for a majority of 1-5 years, failed flame-resistance and water-resistance requirements (1996).

Obviously, further research of used clothing would aid in clearly understanding the moisture transfer in fire fighter protective clothing. Thus far most research conducted on fire fighter protective clothing has exposed the garment directly to the fire. One research area often overlooked in the past is in injuries (such as scalding or burn injuries), which occur outside the fire (Lawson, 1996). Project FIRES reported more fire fighters are killed and injured as a result of physical stress than burns. One reason for this is that moisture (sweat) and metabolic heat become trapped within the garment, causing heat stress (Fornell, 1992). Fornell also reported that higher thermal protective performance (TPP) ratings caused the fire fighters to sweat more and the extra insulation holds the body's heat inside the garment.

More significantly, Torvi et al conclude that additional research would assist in determining the lifetime of turnout gear as well as aid in the evaluation of turnout gear materials (1999). Research shows laundering, high heat and ultraviolet radiation affects turnout gear materials. Test methods and more rigorous preconditioning added in 1997 to NFPA 1971 standards test the durability of turnout gear materials including the moisture barrier. Torvi et al also state a need to agree upon the importance of the factors affecting the durability of turnout gear and design test methods to aid in the evaluation of turnout gear in use (1999).

Purpose

Thus, the purpose of this research was to investigate the failures seen in the moisture barrier of the turnout gear, as noted by the protective clothing industry. The results of this investigation will determine the cause of the failures and lead to development of future tests that will determine whether these failures will occur in other moisture barriers. Specifically, the development of a test method that will predict the failure of the moisture barrier will allow the moisture barrier to be replaced before the fire fighter is at high risk for experiencing heat stress, burn injuries, or hazardous liquids.

Objectives

Specifically, the objectives of this study are:

1. To determine the cause of failure in the protective clothing's moisture barrier layer.

2. To develop a test method to replicate the failures for future testing.

Research Questions

1. Is the failure in moisture barriers caused by ultraviolet light exposure?

- 2. Is the moisture barrier's breakdown affected by abrasion?
- 3. Can the damage in the field be replicated in order to develop a test method that will predict failures?

Limitations

The number of materials available for physical testing limited this research. The

use of limited materials will decrease the number of samples that can be evaluated for

testing purposes. Furthermore, the results of this study may not be related to all moisture

barriers used in fire fighting turnout gear, but only to those evaluated.

Definitions

Fire Fighter: "One who is employed by a fire department to fight fires" (Webster's Dictionary, 1994, p. 480).

Moisture Barrier: "The portion of the ensemble designed to prevent the transfer of liquids" (NFPA 1971, 1997, p. 9).

<u>Neoprene</u>: "A synthetic rubber produced by polymerization of chloroprene and marked by its durability and resistance especially to oil" (Webster's Dictionary, 1994, p. 790).

<u>NFPA 1971</u>: National Fire Protection Association standard on Protective Ensemble for Structural Fire Fighting "specifies the minimum design, performance, certification requirements, and test methods for protective ensembles that include protective coats, protective trousers, protective coveralls, helmets, gloves, footwear, and interface components designed to provide a minimum level of protection for fire fighters against adverse environmental effects during structural fire fighting operations and certain other emergency operations" (NFPA 1971, 1997, p. 6).

<u>Outer Shell:</u> "The outermost layer of the composite with the exception of trim, hardware, reinforcing material, and wristlet material" (NFPA 1971, 1997, p. 8).

<u>PTFE</u>: A microporous membrane with 9 billion pores per square inch. Each pore is approximately 0.2 micron in size and prevents penetration of liquids because of the low surface energy of the PTFE membrane. Evaporated sweat will diffuse through the pores of the membrane carrying body heat with it (Gohlke, D.J., 1980).

<u>Protective Clothing/Protective Ensemble:</u> "Multiple elements of clothing and equipment designed to provide a degree of protection for fire fighters from adverse exposures to the

inherent risks of structural fire fighting operations and certain other emergency operations" (NFPA 1971, 1997, p. 7).

<u>Protective Coat/Turnout Coat:</u> "A protective garment; an element of the protective ensemble designed to provide minimum protection to upper torso and arms, excluding the hands and head" (NFPA 1971, 1997, p.7).

<u>Structural Fire Fighting:</u> "The activities of rescue, fire suppression, and property conservation in buildings, enclosed structures, vehicles, marine vessels, or like properties that are involved in a fire or emergency situation" (NFPA 1971,1997, p. 10).

<u>Thermal Barrier/Liner</u>: "The portion of protective ensemble element composites that is designed to provide thermal protection" (NFPA 1971, 1997, p. 10).

Flexing: "To bend repeatedly" (Webster's Dictionary, 1994, p. 487).

Chapter Two

Review of Related Literature

Turnout gear designed to protect fire fighters has many design components. Each component must meet its own set of protection requirements as well as some composite requirements. The following literature will discuss the fire fighting environment, the fire fighter in the fire environment, the National Fire Protection Association, protective clothing for fire fighters, and the moisture barrier of turnout gear.

Fire Fighting Environment

Fire fighting can be a very dangerous occupation. Potentially, a fire fighter can come in contact with many different hazards that require protection, but the most common hazards are those of direct flame contact and extreme temperatures. When coming in contact with direct flame and extreme temperatures, there are three classifications of fire conditions which fire fighters could possibly face: routine, ordinary, and emergency. Routine fire conditions range in temperature from 68° F to 122° F. These types of fires usually are small, consisting of small objects. An ordinary fire condition ranges from 140° F to an approximate 575° F. The conditions of an ordinary fire are considered more serious than a routine fire. In an ordinary fire condition, the fire fighter may need more protection than his/her protective clothing can provide. When exposed to temperatures of 575° F the fire fighter can only withstand short durations of exposure. A structural fire is typically an ordinary fire condition, which includes those fires or emergency rescues where a structure is involved, such as a building, car, home, etc. (Stull et al, 1996). An emergency condition is where the fire fighter comes in direct contact with the fire a flash or post flashover condition. These conditions could put the

fire fighter at risk of being exposed to temperatures above 1000° F. An example of an emergency fire condition is a flashover. Flashovers occur when the entire room or structure is engulfed in flames.

Other hazards fire fighters may come in contact with include: steam exposure, blood borne pathogens, hazardous chemical exposure, electric shock, and physical hazards such as sharp edges, bursting pipes, and contaminants (Stull et al, 1996). These hazards can be present in many different situations and can differ from situation to situation. Not only are fire fighters exposed to different work environments, they also have requirements of strenuous manual labor such as climbing, carrying heavy loads, and moving quickly.

Fire Fighter in the Fire Environment

The fire fighter in the fire environment is in a very dangerous situation. He or she could easily be faced with an emergency situation where injuries such as burns or heat stress can occur. Often injuries occur because fire fighters may already be overheated and sweating before entering the fire scene. The turnout gear's thermal barrier absorbs sweat and water, which changes the Thermal Protective Performance (TPP). Lawson states that most burns are moisture and compression related, which together accelerate heat transfer (1996).

First-degree burns occur at skin temperatures of about 118°F and second-degree burns occur at temperatures of about 131°F. Exposure to higher temperatures will cause the skin temperature to rise to a critical point where heat losses can no longer be maintained and more serious burns occur. (Lawson,1996). Another common injury factor is that turnout gear provides a delay in heat transfer, and the fire fighter may move

in too close to the thermal zone without realizing the dangerous temperature. Lawson notes, "Once a fire fighter's protective clothing has been heated and the skin temperature has risen to dangerous levels, it is unlikely that a fire fighter can immediately remove the protective clothing and start the cooling process to prevent additional injury" (1996, p. 68).

National Fire Protection Association

The National Fire Protective Association (NFPA) was founded in 1896 by a group of individuals working to improve sprinkler systems. From this beginning the organization has grown to regulate and maintain all aspects of fire safety. Currently they regulate more than 300 standards relating to fire safety (NFPA, 2000).

National Fire Protection Association as an Organization

The purpose of NFPA is to promote the science and improve methods of fire protection and prevention. NFPA's mission, to decrease the problems with fire for all living things, is realized by setting codes and standards, conducting research and providing education. NFPA is comprised of approximately 6000 volunteers from various professions in industry who serve on more than 200 technical committees within NFPA, each with a particular focus. The committee members work continuously throughout the year to set and improve standards. NFPA does not have the power to enforce the standards they set. Because government has adopted many of NFPA's standards, however these standards have become law. Therefore, government has the only power to enforce NFPA's standards. Some of the government organizations, which have adopted many NFPA standards are the Occupational Safety and Health Administration, Veterans Administration, and the Department of Health and Human Services (NFPA, 2000).

National Fire Protective Association 1971 Standards

The first NFPA 1971 Standard was set in 1975 under the title <u>Protective Clothing</u> for <u>Structural Fire Fighting</u> (NFPA, 1975 Edition). Since 1975 the standard has been updated every three to five years. The latest edition of the NFPA 1971 Standard was published in the year 2000. The NFPA 1971 standard sets minimum requirements for elements of the protective clothing ensemble including coats, trousers, one-piece suits, helmets, gloves, hoods, and footwear. The requirements include the design, performance, testing, and certification of firefighters' gear. Usually the standards are updated every five years, but in 2000 it was revised to add the Total Heat Loss Test, tougher preconditioning prior to testing, and a test for thermal conduction of compressed areas such as knees and shoulders. Current tests used to evaluate turnout gear include the Thermal Protective Performance (TPP), Flame and Oven tests, Conductive Compressive Heat Resistance (CCHR) test, Shower Testing, Strength tests, Total Heat Loss (THL) test, Liquid Chemical Resistance test for moisture barriers including water and a Viral Penetration Resistance test for moisture barriers and sealed seams (Lion Apparel, 2000).

Fire Fighters' Protective Clothing

Fire fighters' protective clothing has progressed significantly over the last century. Fire fighter turnout gear has been an issue since the early 1900's when fire fighters wore canvas overcoats and thigh high rubber boots as a mode of protection. As a result of the research in thermal protective clothing supported by the military in the 1940's, fire fighter protective clothing went from canvas and rubber to synthetic and plastic materials. Since the introduction of these materials, many improvements have been made to today's turnout gear (Veghte, 1991). Protective clothing used by fire

fighters is designed to provide "limited" protection from flames, moisture and heat. Protective clothing is not designed to protect the wearer from temperatures above 575° F, even at 575° F it's protects for only short duration exposures. Protective clothing should protect the fire fighter from the different types of fires discussed previously and allow the fire fighter to perform the duties of fighting fires with some comfort and protection. Protective clothing also protects the fire fighter from chemical and biological contaminants and from minor cuts and abrasions (Lawson, 1996).

The Protective Clothing Ensemble for Fire Fighters

There are 6 elements included in the fire fighters protective ensemble. These are a helmet, a hood, turnout coat and pants, gloves, footwear, plus breathing apparatus. Each of these items has different functions, which aid in the protection of the fire fighter (SAFER, 1994). The helmet is used to protect the face and ears from physical and thermal hazards. The helmet is composed of an outer-shell, an impact cap, suspension system, trim, a face shield, a chinstrap, and ear covers (SAFER, 1994). The second item of the protective ensemble is the hood. The hood protects the fire fighter's ears, neck, and face from exposure to extreme heat. The hood is designed to protect the head and neck area not protected by the helmet (SAFER, 1994) or the coat. The turnout coat is the third item and provides "limited thermal and physical protection to the upper torso and arms (excluding hands and head)" (SAFER, 1994). The NFPA (1971) requires that there be three layers in the turnout coat -- the outer shell, moisture barrier, and thermal barrier. Other items included in the design of the coat are reflective trim, closure systems, and wristlets (SAFER, 1994). The turnout pants are designed to provide the lower torso and legs with "limited thermal and physical protection" (SAFER, 1994, p. 16). The

components of the pants are the same as the coat, consisting of the same three layers, reflective trim and closure systems (excluding wristlets) (SAFER, 1994). Stull et al (1996) describe the ensemble of firefighters' protective clothing as being either a coat and pant ensemble or a single coverall. The collar and wristlets of the coat protect those interface areas not enclosed by the coat. Both the coat and pant ensemble or coverall are designed for quick and easy entry. The reflective trim allows for visibility (Stull et al, 1996). Gloves provide "limited thermal and physical protection" (SAFER, 1994, p.16), to hands and wrists. Gloves also protect from blood borne pathogens, and some fire ground liquid chemicals. The gloves are made with an outer shell, a moisture barrier, and a thermal liner (SAFER, 1994). The footwear is the seventh item of protective clothing for the fire fighter. Footwear provides "limited thermal and physical protection to the wearer's feet and ankles" (SAFER, 1994, p. 18). The footwear consists of an outer shell, a steel shank, a thermal liner, and steel toes (SAFER, 1994).

Layers of the Turnout Gear

There are many different materials used in today's firefighters' protective clothing. However, the primary criterion is that all the materials used must be flame resistant. The most common fibers used in the material of turnout gear are aramids (Nomex®, Kevlar®), and PBI. These fibers are often blended together in a textile for the purpose of enhancing performance characteristics and/or creating different weights depending on the end user's environment. The first layer of protection in the turnout gear is called the outer shell. The outer shell provides protection against flame and heat, wear and abrasion (Fornell, 1992). It resists ignition for short periods of direct flame contact (Lawson, 1996).

The materials typically used in the outer shell are products made of aramid fibers or PBI. One outer shell material, made from meta- aramid fiber, is Nomex®. Nomex® is used as an outer shell material because it is flexible, sturdy, lightweight, and protects from heat and flame. The most common outer shells of Nomex® available are 7.5 ounces per square yard and 6 ounces per square yard. Some fabrics used in the outer shell maybe constructed with a woven rip-stop weave, which prevents the continuation of a rip or tear (Fornell, 1992). Another outer shell material is PBI. PBI is blended with Kevlar® fibers, which are from para-aramid fiber. Kevlar® is used in the outer shell for its strength, flexibility, and high heat/flame resistance. PBI also is woven with a rip-stop weave and is available in 7.5 ounces per square yard or 6 ounces per square yard. Others combine the characteristics of both Nomex® and Kevlar® fibers (Fornell, 1992). The blends are typically Kevlar® rich comprising 60% of the fiber weight

The moisture barrier is typically the middle layer, which is made of a urethane, PTFE or Neoprene coated textile or laminate consisting of a film, adhesive and substrate of high heat resistant fibers (Stull et al, 1996). The moisture barrier is used to prevent water from soaking through the entire garment. The moisture barrier seams are sealed with seam tape then the entire barrier is sewn to the thermal barrier, the third layer of protection forming a liner system that provides insulation.

The thermal barrier insulates the fire fighter during high heat loads. The insulating quality of the thermal barrier is dependent on air spaces within the fabric and the heat transfer properties in materials used to make up the thermal barrier (Lawson, 1996). The thermal barrier is constructed of an insulating material, which retards heat flow through the garment and is typically made of a nonwoven textile of Nomex®

Kevlar® blend (Lawson, 1996). The nonwoven structure is quilted to a lightweight woven fabric that is also flame resistant (Stull et al, 1996).

Moisture Barrier

Some moisture barriers prevent liquid and impermeable vapor from reaching the skin while others prevent liquid from reaching the skin but allow the transfer of permeable vapor (Lawson, 1996). Torvi et al (1999) report that moisture transfer has a significant effect on the heat transfer through these garments, and hence, the garment's comfort performance. A breathable moisture barrier helps reduce heat stress and the possibility of steam burns (Torvi et al., 1999).

Materials used in the Moisture Barrier

Water can interact with various fibers in different ways. It can be absorbed, adsorbed, wicked, or repelled. To provide protection from wetting, a film or coating may be added to the fabric. These films or coatings may be composed of many different treatments, all of which help prevent water from passing through to the wearer, while allowing the body to breathe (vapor from evaporation escape). According to Fornell (1992), there are two types of moisture barriers-- impermeable and expanded membrane polytetraflouroethylene (PTFE) liner. The impermeable barrier is coated with a fireretardant neoprene on either poly-cotton or Nomex rip-stop fabrics. Gore-Tex® and Tetratex® are two types of expanded membrane PTFE liners (Fornell, 1992). The moisture barrier consists of two parts, a film or coating which is applied to a substrate that is either woven or nonwoven. The film can be either semi-permeable or impermeable. There are many different breathable moisture barriers such as

CROSSTECH[®], Vapro[™], Breathe-Tex[™], and ComfortZone[®] (WFR, 1999), AquaTech[™], Stedair[®] 82 etc.

Moisture Barrier Systems

A moisture barrier system is how the film is constructed to allow moisture vapor to flow through the garment. The three basic film systems used in moisture barriers include microporous, monolithic, and bi-component. The microporous systems have minute micro size openings or pores throughout the polymeric membrane, which allow moisture vapor to pass through. The fabric can be either hydrophilic or hydrophobic (Gore, 1998). The second type of moisture barrier fabric is the monolithic. The fabric consists of a thin coating with no passages for true air or moisture to penetrate. The monolithic fabric can be either neoprene coated, particulate filled, or polyurethane-based coated (Gore, 1998). The third fabric is the bi-component. Gore (1998, p. 1) defines the bi-component as "that which truly combines the performance attributes of the microporous and monolithic technologies."

Lawson (1990) recognizes the three basic systems as polymer membranes used in breathable textiles as microporous films and coatings, hydrophilic films and coatings, and combined microporous and hydrophilic layers. Microporous membranes allow vapor to pass through the permanent, vapor-permeable pore structure. Hydrophilic membranes carry vapor through the garment by a molecular mechanism, which is a process of absorption, diffusion, and desorption.

There are three different applications used in hydrophilic polyurethane moisture barriers according to Lomax (1990). The first is a nonporous coating on a base fabric. The coating can be either a one- or two- component polyurethane, which is applied to the

base fabric by a normal direct, or a transfer coating process. When this process is used for microporous coatings, the coating appears white due to the refracted light through the porous surface. The second type is a solid polyurethane layer used on microporous polyurethane and PTFE membranes. Solid polyurethane layers are used to seal the surface pores and reduce chances of contamination from various substances such as soap and salt residues, particulates air-borne dirt, and surfactants, which could affect the breathability or waterproofness of the film or coating (Mooney, 1985). The third application of hydrophilic polyurethane is the use of adhesives to laminate the breathable membrane to a base fabric. This process reduces the loss of breathability, which occurs during laminating. The majority of hydrophilic polymers are not suitable for use as a permanent, flexible fabric. They are too sensitive to liquid, either dissolving or not withstanding normal use (Lomax, 1990).

Microporous membranes are manufactured by stretching the product. The stretching process creates micro-cavities in the film or coating. PTFE and polyolefins are examples of microporous membranes (B.F. Goodrich, n.d.). Monolithic membranes are manufactured by casting a film onto a fabric by lamination. Because of this technique there are no holes. Monolithic membranes are waterproof, whereas microporous membranes only resist liquid. Surfactants used on microporous membranes may cause the structure to leak, whereas monolithic membranes are unaffected by surfactants. Microporous membranes have a low level of pressure at which water can enter the structure. A monolithic membrane requires high pressure to allow water to enter the structure (B.F. Goodrich, n.d.).

According to Krishman, (1993), breathable coatings possess good moisture vapor transmission, tape sealability, wet and dry abrasion resistance, durability to multiple washings and dry cleanings, good low temperature flexibility, and good hydrostatic resistance. Microporous systems possess good moisture vapor transfer, lack adhesion and abrasion resistance, and have poor dry cleaning properties. Hydrophilic systems have a lower moisture vapor transfer, good adhesion, tape sealability and abrasion resistance (Krishman, 1993).

Aldan Industries (n.d.) categorized the moisture barriers systems sold today into three groups: microporous polyurethane; cast coated, crosslinked hydrophilic polyurethane; and stretched Teflon® with hydrophilic coating. The microporous polyurethane allows water vapor to pass through while preventing water from entering. The cast coated, crosslinked hydrophilic polyurethane allows water vapor to pass through by diffusion. The stretched Teflon® with hydrophilic coating allows water vapor to pass through by microporous film. The stretched Teflon® also contains a hydrophilic layer. *Moisture and Heat Transfer in Turnout Gear*

Protective clothing for fire fighters decreases heat and moisture flow from the fire scene to the wearer; it also decreases heat and moisture flow from the wearer to the fire scene. This prevents the wearer from quickly losing body heat, which causes a rise in body core temperature. According to Lawson (1996, p. 6), "The body may become heat stressed which activates the sweating process in an attempt to restore a normal body temperature." Because protective clothing does not allow the flow of liquid, limited cooling occurs and sweat from the body cannot evaporate easily. When the thermal

barrier absorbs sweat, it could decrease the barrier's insulating properties (Lawson, 1996).

When sweating, a fire fighter is in danger of steam and scald burns at temperatures as low as 212°F (Veghte, 1987). When temperatures rise within the garment, the moisture from sweat and leaking becomes trapped inside the gear and heated to temperatures that may cause serious burns. Moisture collected on the outer shell will evaporate and cause cooling which carries heat away from the clothing. Moisture trapped in the thermal barrier may decrease the TPP of the garment. TPP measures the amount of protection from heat transfer through protective clothing layers in conditions close to those of a flashover situation. According to Fornell, "TPP is used to quantitatively evaluate fabrics for thermal protection" (1992, p. 106). In 1985 Project FIRES, a program started by NASA to attempt to address the problem of heat stress, discovered thermal protection alone should not be the only concern. Because higher TPP ratings cause more sweating and heat is held inside the garment, it is important to note that TPP tests are performed dry and water transfers heat more quickly (Lawson, 1996).

Conductive heat transfer occurs when water is 190°F 21 times faster than in air at temperatures as high as 200°F (Bennet, et al, 1974). Water in a garment will produce higher heat transfer (inward) rates, so the rate may be affected by an increase in the moisture evaporation rate. The turnout gear is more conductive to heat when water or other fluids are trapped in interstices or voids. In extreme instances where water is at high temperatures, safety of the wearer becomes an issue.

Hot water vapor and steam are also safety issues for the wearer when temperatures are extreme. As condensation of steam reaches skin of cooler temperatures,

burns will occur. For water to evaporate, heat must be present. The release of heat causes steam to be transformed to a liquid condensation. When the body is exposed to heat in "un-withstand able" rates, sweating and heat exhaustion could occur. Once the burst of instant heat reaches the body, burns may also occur. Skin can be damaged from heat at approximately 111°F; therefore the moisture barrier must protect the fire fighter from these conditions (Watkins, 1995).

Further, "moisture barriers that allow the flow of moisture vapor have a body core temperature approximately 1.8° F less" than a moisture barrier that does not allow the flow of moisture vapor (Lawson, 1996, p. 7). Research by Huck (1987) shows that a change in body core temperature of 1.8° F can be critical. Veghte (1988) claims that body core temperature of fire fighters can commonly be as high as 101° F. Huck (1987), however, notes that at a body core temperature of 102° F, the body begins to lose efficiency and medical problems begin to occur. Long periods of exposure to high temperatures will cause a rise in skin temperature when heat loss, which protects the skin, is no longer maintained and burns occur. Blood flow, thermal radiation of the skin's surface, and heat loss from sweat affects the skin's heat loss (Lawson, 1996). According to Veghte (1987, p. 316), "Fire fighters become susceptible to steam or scald burns, once sweating begins". Although some moisture barriers will allow the transfer of water vapor, they do not allow the flow of liquid.

Burns and scalding occur when temperatures within the protective clothing are below boiling point (212° F) and moisture is present from sweat and areas where leaking may occur (Lawson, 1996). Lawson suggests that the reduction and control of moisture inside protective clothing reduces fire fighter burn injuries (1999). Stull demonstrates

that moisture's effect on the performance of fabric depends on the location and amount of moisture (n.d.). The moisture barrier keeps the thermal barrier dry from outside sourced liquids. When the thermal barrier becomes wet from sweating and/or leaking, the insulative value is reduced and its weight is increased. The moisture barrier also prevents air from penetrating to the thermal barrier, which can reduce the insulative value. (Veghte,1991). Lawson concludes that improvements should be made on the reduction and control of moisture inside the protective clothing (Lawson, 1996).

Rossi and Zimmerli, Zimmerli and Weder, and Mäkinen, Ilmariner, Griefahn, and Künemund have studied moisture transfer in turnout gear. Rossi and Zimmerli examined moisture's influence on heat transfer in the turnout gear ensemble and the influence of the moisture barrier (1996). Fourteen turnout gear ensembles were exposed to a simulated humid environment and radiant, convective, and contact heat. Rossi and Zimmerli found breathable barriers provide more protection than impermeable coated materials when exposed to radiant or convective heat and water is present. Zimmerli and Weder developed a device, which replicates a sweating torso to measure thermal protection and comfort of turnout gear for fire fighters (1997). The sweating torso stimulates the heat and sweat produced by humans and can be exposed to a fire fighter's environment to predict the physical environment of fire fighters. Mäkinen et al. (1996) measured physiological stress of turnout gear with and without a microporous membrane moisture barrier. This study found thermal stress in both ensembles. Mäkinen et al also found more sweat in the underclothing, and higher physical exertion and thermal discomfort in the garment with the membrane (1996). Rossi and Zimmerli's (1996) study on fire fighters' clothing reported that exposure to radiant heat caused a decrease in water vapor

permeability. Fabrics with a lighter outer-shell showed a greater decrease in water vapor permeability than those with a heavier outer-shell.

Torvi et al note that moisture transfer is difficult to describe due to the various conditions fire fighters face (1999). Moisture transfer affects heat transfer in the garment and its performance. Torvi et al also indicate more research would aid in understanding the moisture transfer in turnout gear. Further, improvements needed in test methods would aid in the evaluation of the amount of protection offered in turnout gear, with emphasis on the moisture transfer that occurs during fire fighting tasks. Specifically, Torvi et al state the need for development of techniques, which apply to moisture in the garment and replicate actual usage (1999).

Durability and Useful Lifetime of Turnout Gear

There are many factors which affect the lifetime of turnout gear such as film and fiber type weight and type of weave of the fabric, frequency of use, number and types of repairs, cleaning procedures used, improper storage to light, types of work performed by the wearer, and exposures to extreme heat, soot, bearing hazardous materials, and ultraviolet radiation (Torvi et al, 1999).

According to Torvi et al, little research has been conducted into the performance of used turnout gear (1999). However, it is known that turnout gear doesn't last forever. One aspect of turnout gear fire fighters and researchers have not been able to determine is the useful lifetime of the gear. If a rip or hole appears in the outer shell or failure is seen anywhere on the outer portion of the gear, the fire fighter can assume it is time to repair or replace his or her gear. The fire fighter can only determine the lifetime of the gear with an evaluation of the outer shell and thermal liner, looking for holes or wear areas.

Manufacturers cannot predict the expected lifetime of the garments when exposed to ultraviolet radiation, heat exposure, or different cleaning and storage procedures, and usages (Torvi, et al, 1999).

<u>Ultraviolet Radiation and Heat Flux</u>. Several researchers have studied the effects of ultraviolet radiation on protective clothing. Day, et al exposed fabrics used in turnout gear to a xenon arc Weather-Ometer and heated oven (1988). Fabrics were examined before and after exposure. The researchers concluded exposure to light and heat reduced the strength of the fabric. Light and heat did not affect flame resistant or TPP properties of the fabrics. Rossi and Zimmerli examined effects of high heat fluxes on turnout gear fabrics (1996). Their study showed that the moisture barrier, the most important component of turnout gear, began to degrade as a result of heat exposure.

Abrasion. Vogelpohl conducted research on 20 garments that had been used for one or more years in fire fighting or training programs (1996). Vogelpohl evaluated TPP, flame resistance, wear resistance, tear resistance, abrasion resistance, water resistance, tensile and seam strength, ultraviolet degradation, zipper operation resistance, and retroreflectivity. The results were compared with tasks and length of time the garments had been used. Vogelpohl's (1996) study found a decrease in water resistance in all the moisture barriers over time. The microporous membrane of the moisture barrier loses its protective properties with wear and abrasion. The wear and abrasion takes place when the three layers abrade each other during the movement of the wearer (Gore, 1996). Failures in the moisture barrier can lead to heat stress for the fire fighters (Slater, 1996). Vogelpohl (1996) suggests that failure results of moisture barriers found in waterresistant tests, water permeability, high range resistance, and penetration resistance to

synthetic blood, may be related to abrasion. She (1996) also recommended that more indepth tests be done on the different moisture barrier fabrics seen in today's protective clothing turnout gear.

<u>Cleaning.</u> Researchers Loftin and Mäkinen have studied the effects of laundering on materials used in turnout gear. Loftin (1992) conducted numerous industrial launderings on turnout gear materials and compared flammability, TPP, abrasion resistance, and strength tests. Mäkinen evaluated the effects of laundering and wear on fabrics used in turnout gear (1992). He found wear and laundering were more significant than laundering alone. Mäkinen suggests when testing the effects of laundering on turnout gear, fabric wear should be included in testing.

Summary

Over the past several years a great deal of research has been done to improve fire fighters' protective clothing. However, little research has been conducted on used protective garments. Torvi et al (1999) conclude that more research is needed to examine the factors that affect protective clothing in use. Research is continuing in the protective clothing industry for new developments and improvements for protection and comfort, but additional research would assess the longevity of fire fighters' clothing, particularly in relation to protecting the body from heat, stress steam burns, and hazardous liquid penetration.

Moisture transfer has a significant effect on heat transfer through the protective ensemble. Veghte (1987) and Slater (1996) both show that the moisture barrier plays a major role in protecting the fire fighter from scald burns and heat stress. An investigation of the failures seen in the moisture barrier will lead to the development of future testing

methods for moisture barriers and will allow for improvements in the protective ensemble to protect the fire fighter. An investigation of the failures also will allow for future tests to be conducted on moisture barriers to predict degrading in the moisture barrier.

Chapter Three Methodology

The purpose of this research was to investigate the failures seen in the moisture barrier of the turnout gear, as noted by the protective clothing industry. This chapter first describes the research design and the methodology that will be used in this study. Second, the sample selection and preparation process will be discussed. The third section will describe the instruments and measurements. Finally, a description of procedures and data analysis will be discussed.

Research Design

A quantitative research design was used throughout this study to allow for the collection of data in a numerical form. The method used was a quasi-experimental design. Moisture barrier samples were chosen and tested without randomization of the samples. The fabric samples were placed in controlled environments and evaluated. Multiple replications of each condition were evaluated and compared to a control sample.

Evaluation of Failed Garments in the Field

A preliminary investigation of failure observed in the field was conducted. Five fire fighting turnout pants, where failure was suspected, were examined. Breathe-Tex® the moisture barrier of the garments were separated from the thermal liner for assessment. The evaluation of the pants consisted of a visual examination of the moisture barrier using stereo and compound microscopes.

Sample

The product under investigation is one component of the turnout coat ensemble, the moisture barrier. In this study, nine different moisture barriers were used, which represent those moisture barriers found on the market or in use today. The moisture

barrier fabrics are: NeoGuard[™], AquaTech[™], ComfortZone[™], CROSSTECH® on E-89 Type 2C, CROSSTECH® on Pajama Check, Breathe-Tex®, RT 7100 PTFE Type 3A, 2000 Stedair® 2000 and Stedair® 82. The following table describes the different moisture barriers used in the study:

Sample	Type of Film or Coating	Type of Substrate	Fabric Weight (oz/yd²)	Fabric Thickness (mils)
A-D and N Three-piece Ensemble	Urethane Film	E-89 17.8		120
Е	PTFE Film	Pajama Check	4.7	20
F	PTFE Film	E-89	3.7	30
G and Y (Breathe- Tex®)	Urethane Film	E-89	3.7	30
H	Urethane Film	E-89	4.9	30
Ι	Neoprene Coating	Polyester/ Cotton	12.1	20
J	Urethane Film	E-89	4.3	30
K	Urethane Film	E-89	5.0	30
L	PTFE Film	Vilene	3.8	30
М	Urethane Film	Vilene	4.1	30
O, P and W Three-piece Ensemble	PTFE Film	Pajama Check	18.5	110
Q and R Three-piece Ensemble	e-piece Urethane Film E-89		17.7	110
S and T Three-piece Ensemble	PTFE Film Vilene 17.7		17.7	110
U and V Three-piece Ensemble	Urethane Film	Vilene	17.7	110
X Three-piece Ensemble	PTFE Film	Vilene	17.7	110

Table 3.1: Description of Moisture Barrier Samples

Treatments and Procedures

Sample Preparation

The specimens were divided into three groups and preconditioned according to NFPA 1971 2000 Edition. Group 1 consisted of two unwashed three-piece ensembles Sample A and C. Group 2 consisted of two pre-washed three-piece ensembles Samples B and D and N-X. Group 3 consisted of nine different pre-washed, heat exposed, single moisture barriers Samples E-M and Y. The three-piece ensembles used in Groups 1 and 2 were constructed of the same components found in today's fire fighter turnout gear. Groups 1 and 2 were exposed and evaluated to allow for two replications of each group and Group 3 was exposed and evaluated to allow for four replications of each single moisture barrier. All specimens were cut into 6 1/2" x 9 1/2" rectangles. The size of the specimens was dictated by the dimensions of the 6 1/2" x 9 1/2" sample holder used for exposure treatment.

<u>Group 1.</u> Unwashed/Three-Piece Ensemble. The first grouping consisted of two samples. These two samples were used to construct the protective ensemble as worn by the fire fighter. A 6 1/2" x 9 1/2" Aralite® thermal liner test piece was the first layer. The thermal liner was placed with the face cloth of the fabric facing upward. The second layer was the Breathe-Tex® moisture barrier. The moisture barrier was placed directly under the Aralite® liner, with the film side of the moisture barrier facing up toward insulative batting. The third layer was a 6 1/2" x 9 1/2" PBI outer shell. The outer shell was placed over the moisture barrier.

<u>Group 2.</u> Pre-Washed/Three-Piece Ensemble. The second group consisted of two samples. These two samples were preconditioned according to the NFPA 1971

Standard on Protective Ensemble for Structural Fire Fighting, 2000 Edition 6-1.2, standard. Samples were subjected to five cycles per American Association of Textile Chemist and Colorist (AATCC) 135, using Machine Cycle 1, Wash Temperature V $(60\pm 3^{\circ}C [140\pm 5^{\circ}F])$ and Drying Procedure Ai: Tumble Cotton sturdy. A 1.82 Kg ± 0.1 Kg (4.0 lb \pm 0.2 lb) load was used, without a laundry bag. Two moisture barriers were stitched together with film or the coating side facing each other prior to treatment. This protected the film or coating from direct exposure to cleaning procedures or burrs. This was done for all samples being used. Following preconditioning, Group 2 was also used to construct the protective ensemble as worn by the fire fighter. A 6 1/2" x 9 1/2" Aralite® thermal liner test piece was the first layer. The thermal liner was placed with the face of the fabric facing upward. The second layer was the moisture barriers, which were selected from Table 3.1. The moisture barrier was placed directly under the Aralite® liner, with the film side of the moisture barrier facing the batting of the thermal liner. The third layer was a 6 1/2" x 9 1/2" PBI outer shell. The outer shell was placed over the moisture barrier.

<u>Group 3.</u> Pre-Washed/Heat Exposed/Single Moisture Barriers. The remaining nine samples were single moisture barriers, which are described in Table 3.1. Each moisture barrier was preconditioned following NFPA 1971 Standard on Protective Ensemble for Structural Fire Fighting, 2000 Edition 6-1.2. Samples were subjected to five cycles per AATCC 135, using Machine Cycle 1, Wash Temperature V ($60\pm 3^{\circ}$ C ($140 \pm 5^{\circ}$ F)) and Drying Procedure Ai: Tumble Cotton sturdy. A 1.82 Kg \pm 0.1 Kg (4.0 lb \pm 0.2 lb) load was used, without a laundry bag. Specimens were exposed to the NFPA 1971 Standard on Protective Ensemble for Structural Fire Fighting, 2000 Edition 6-1.5

Convective Heat Conditioning Procedure for Helmets, Gloves, Footwear, Moisture Barriers, Moisture Barrier Seams, Labels and Trim. The oven test temperature was stabilized at $140^{\circ}C + 6/-0^{\circ}C$ ($285^{\circ}F + 10/-0^{\circ}F$) and the test exposure time was 10 minutes, +15/-0 seconds. Two moisture barriers were stitched together with the film or coating sides facing each other prior to treatment. This protected the film or coating from direct exposure to cleaning procedures or burrs in washer/dryer and oven testing. This was done for all samples being used. The procedure was repeated and the samples were cut into 6 1/2" x 9 1/2" rectangles. The following table summarizes the pretreatment conditions of the specimens.

Table 3.2: Pretreatment Condition

Group	Sample	Conditions	Replications
1	A and C	Unwashed	0
2	B and D, N - X	Pre-washed and dried	5
3	$\mathrm{E}-\mathrm{M},$ and Y	Pre-washed and dried 5 times and heated to 285°F for 10 Minutes	2

Exposure Treatment

All samples were exposed to an ultraviolet light source. Groups 1, 2, and 3 were exposed to a Carbon Arc Fade-o-meter, whereas only Groups 2 and 3 were exposed to natural sunlight exposure.

Instrumental. Carbon Arc Fade-o-meter. The Carbon Arc Fade-o-meter was used according to AATCC Test Method 16-1998, Option A. This test method allowed for determining the effects of ultraviolet light on the moisture barriers. The Enclosed Carbon Arc transmits 275 to 370 nanometers of wavelengths. Thirty-six samples were exposed, in 20-hour increments, to the Enclosed Carbon Arc at ambient temperatures.

The specimen holders placed in the Fade-o-meter were 6 1/2" X 9 1/2". The following table summarizes the conditions to which each sample was exposed to the Carbon Arc Fade-ometer.

Sample	Fabric Exposed to Carbon Arc Fade-ometer	Fabric Directly Exposed to Light Source
A and C	Unwashed three-piece ensemble including outer shell, moisture barrier and thermal barrier	Thermal Barrier
B and D	Pre-washed three-piece ensemble including outer shell, moisture barrier and thermal barrier	Outer Shell
E-H and J-M	Pre-washed single moisture barrier	Film side facing the light source
I	Pre-washed single moisture barrier	Coating side facing the light source

 Table 3.3: Summary for Carbon Arc Fade-ometer Exposure Conditions

<u>Sunlight</u>. A natural sunlight laboratory, Q-Panel Laboratory, located in Homestead, Florida was used to expose samples. Two hundred fifty two samples were exposed to natural sunlight for fourteen weeks, according to ASTM G7 test method. Pre-washed three-piece ensembles from Group 2 were exposed to sunlight as well as single moisture barriers from Group 3. Each week one sample of each type of moisture barriers was removed and returned to the University of Kentucky Textile Testing Laboratory for evaluation. Moisture barrier types and codes are described in Table 3.4. Temperature and relative humidity was recorded throughout each day of exposure. The total number of days each sample was exposed was also recorded. Table 3.4 summarizes the conditions to which each sample was exposed during the natural sunlight treatment.

Sample	Fabric Exposed to Natural Sunlight	Fabric Directly Exposed to Light Source
B, P, R, T, V, W, and X	Pre-washed three-piece ensemble including outer shell, moisture barrier and thermal barrier	Thermal Barrier
N, O, Q, S, and U	Pre-washed three-piece ensemble including outer shell, moisture barrier and thermal barrier	Outer Shell
E, F, G, L, M, and Y	Pre-washed single moisture barrier	Film side facing the light source

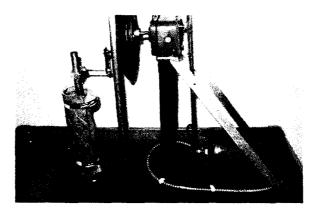
Table 3.4: Summary for Natural Sunlight Exposure Conditions

Flexing Treatment

A pilot test device was used to develop a procedure to flex the samples. The device allowed for consistent flexing of all specimens except for two of the three-piece ensembles. In the initial pre-testing of the samples, flexing was not used as a treatment. Due to the length of time required to degrade the specimens, flexing was added to accelerate the process of degradation. Two of the three-piece ensembles, one from Group 1 and one from Group 2 were not flexed to allow for comparison of the flexed and unflexed specimens. This was done to determine whether flexing affects the degradation of the moisture barrier. The flexing procedure was chosen because the fire fighter is flexing the fabrics while the turnout gear is in use. The flexing procedure closely resembles the bending at the knee and elbow areas of the turnout gear. The pilot test device utilized the AATCC Wrinkle Tester. The pilot test motorizes the AATCC Wrinkle Tester to allow for synchronization and stabilized flexing for all specimens being flexed. The flexing device was designed to hold the sample size compatible with the instruments used for ultraviolet light exposure. The rod in the center of the device is attached to the motor.

When the motor is in operation the rod is moving in an up and down motion, creating the flexing. Two specimens are clamped to the center rod at one time, allowing for . accelerated testing. While flexing the three-piece ensembles, the outer shells were facing the center rod and the thermal liner was on the outer side of the flexing device. While flexing the single moisture barriers, the substrate side of the barrier was facing the center rod. To allow for two specimens to be flexed together, a three-piece ensemble was flexed with a single moisture barrier or alone, and two or one single moisture barriers were flexed together. While two specimens were being flexed together, an overlap of the two was necessary. The samples were flexed for 5 minutes (approximately 300 flexes) prior to each 20-hour increment of ultraviolet light exposure. An illustration of the flexing device can be seen in the following Figure 3.1.

Figure 3.1: Pilot Flexing Device



Performance Measurement

Following treatment procedures, all samples were evaluated for microscopic appearance and water penetration resistance to assess the visual appearance and performance of the moisture barrier.

<u>Microscopic</u>. Microscopic evaluations were conducted on all specimens prior to exposure to assess the quality of the moisture barriers before exposure. Microscopic

evaluations were performed following each 20-hour increment of instrumental exposure. Two types of microscopic evaluations were conducted. First, a stereo evaluation was conducted using a Zeiss Stereo Microscope where magnification ranged from 7X-35X. This enabled the magnification of surface appearance. The second evaluation was conducted on a Zeiss Compound Microscope where magnification was 100X, which enabled the researcher to take a closer look at apparent flaws or degradation.

Hydrostatic Water Penetration Resistance Test. To determine product failure a Water Penetration Resistance test was used. The test measures the water pressure required to penetrate through a fabric in pounds per square inch (psi). The Water Penetration Resistance test was conducted using a W.L. Gore and Associates, Inc. Hydrostatic Water Penetration Resistance Tester. The test used a modification of NFPA 1971, 1997 Edition 6-27. 4.2 Procedure B; at 0.07 Kg/cm² [1 psi] for five minutes in accordance with Method 5516, "Water Resistance to Cloth: Water Permeability, Hydrostatic Pressure Method," of Federal Test Method Standard, 191A, Textile Testing Methods. The Carbon Arc Fade-ometer samples were exposed to 2 psi for 2 minutes, whereas, the natural sunlight samples were only exposed for 30 seconds at 2 psi. This modification was decided to vary the time constraints of testing. Only single layer moisture barriers were tested, including the moisture barriers in the three-piece ensembles. When testing the water penetration resistance of the three-piece ensembles, the moisture barriers were removed from the ensemble to be tested and returned to the ensemble for exposure to the ultraviolet light. Specimens were placed on the Hydrostatic Tester with the film or coating side face down. The samples were tested prior to ultraviolet light exposure at 2 psi for 2 minutes. This modification of the Hydrostatic

Water Penetration Resistance Test required higher psi in a shorter period of time than the NFPA procedure. This allowed for the samples to be tested at a faster pace. Specimens were tested following each 20-hour increment of instrumental exposure and after each week of natural sunlight exposure. Once a specimen failed the Hydrostatic Water Penetration Resistance Test, it was removed from the sample holder and replaced with an unexposed specimen to allow for replication. Specimens were tested after the first initial flexing as well as after exposure to direct sunlight.

Method of Data Analysis

The data from each individual test were examined, evaluated, and recorded after exposure treatments. The data were analyzed using statistical measures and a statistical software package. Descriptive statistics were used for comparison of replications within samples. A General Linear Model was conducted to test within the samples and a Mauchly's Test of Sphericity to test for significant differences. The results from the different exposures were compared to allow for the development of a test method that utilizes the best light source. The results were reviewed to determine how well they answer the research questions of this study.

Chapter Four

Results and Discussion

This study was conducted to investigate the failure seen in the moisture barriers of fire fighter's turnout gear, as noted by the protective clothing industry. Nine different moisture barriers were exposed to artificial light and natural sunlight. The effect of light on the moisture barriers was evaluated visually using stereo and compound microscopes and for performance by testing for water penetration using the Hydrostatic Water Penetration Resistance Test.

Prior to physical testing, the moisture barriers were examined visually to assess their quality and initial appearance. Moisture barriers were also tested for water penetration resistance. To ensure accuracy of testing, only samples which passed the initial water penetration resistance test were used.

Three treatments of moisture barriers included exposure to instrumental light in the Carbon Arc Fade-ometer and to natural sunlight and also involved flexing the moisture barriers to simulate flexing and surface abrasion. Microscopic evaluations and Hydrostatic Water Penetration Resistance Tests were performed to assess degradation and failure.

This chapter will discuss the results of the evaluations. The statistical analysis of the data also will be discussed in this chapter. The analysis of the data was used to determine if ultraviolet light exposure and abrasion had a significant effect on the failure of the moisture barrier and whether the test can be replicated to predict future failures.

Examination of Failed Garments from the Field

In the preliminary investigation of the moisture barrier problem, hundreds of garments from the field were inspected. The garments had been in use from 1-5 years in a range of environments including the hot humid conditions of New Orleans or Florida to the cold and winds of Chicago. Of these garments, 5 were selected by the researcher to conduct a closer examination. The 5 garments were selected because they represented the type of degradation seen in the field, with a history of 1-3 years of use.

All garments contained Breathe-Tex® moisture barrier. The moisture barriers of the garments were examined visually and microscopically. The moisture barrier's film was originally gray in color but in the field garments the moisture barrier film became a blue/green color. The moisture barriers also demonstrated severe cracking and flaking of the film. When viewed under the compound microscope, lighter areas showed thinning of the film or a complete loss of film. For all five garments the most severe film damage was represented by color change, cracking, and flaking had occurred in areas where the thermal liner was exposed to light and/or the areas most susceptible to abrasion during continued use. For example, the moisture barrier showed evidence of severe degradation in the seat and waist areas of the pants. This may be due to the habits of use, storage and cleaning; for example when the fire fighter's turnout gear is not in use the pant are pulled down over the boots, which exposes the seat and waist of the thermal barrier to the light source. The moisture barriers in the lower pant sections had not experienced the color change and damage that was apparent in the sections of pants that garment.

The preliminary examination of field garments appeared to show a direct correlation to light as a contributing factor in the degradation of the moisture barrier. For

instance, the pants showed no degradation in the lower legs of the pants but severe degradation in the top, especially the sections typically draped over the boots during storage between uses.

Light degradation was thought to be a major factor in the damage seen in the moisture barriers but damage had been seen in garments exposed to limited sunlight but exposed to indoor or filtered light during storage. The result of the preliminary examination of product failure led to the selection of two light exposure treatments, instrument ultraviolet and natural sunlight. Figure 4.1 illustrates the damage seen in the moisture barrier.

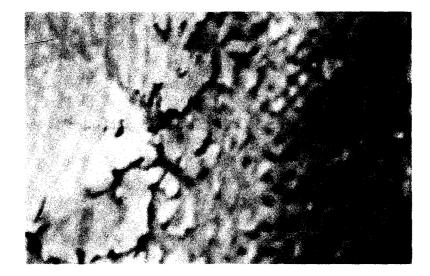


Figure 4.1: Degradation Observed in Failed Garments from the Field

Exposure to Instrumental Light – Carbon Arc Fade-ometer

Initially, samples were exposed to an Atlas Carbon Arc Fade-ometer. The Fadeometer is an artificial light source that uses ignited carbon rods to transmit light in the 275-370 nanometer range and approximates ultraviolet light, in ranges from 250 to 400 nanometers. This phase of testing was conducted to determine if wavelengths of ultraviolet light contributed to or caused degradation of the moisture barrier. The samples were exposed in 20-hour increments at ambient conditions. After 20 hours of exposure to the Fade-ometer, the samples were subjected to a Hydrostatic Water Penetration Resistance Test, with water pressure set at 2 psi for 2 minutes exposure, to determine whether they leaked. If they passed the test, samples were exposed for another 20 hours. All samples were exposed in 20-hour increments until failure occurred during the Hydrostatic Water Penetration Resistance Test. Once failure occurred, the failed sample was pulled from the chamber and replaced with another replication of the sample. Exposure data was reported as the number of hours of Carbon Arc exposure required before failing the Hydrostatic Water Penetration Resistance Test. Visual observations were recorded before and after exposure using stereo and compound microscopic evaluations.

Phase 1

The first phase of testing began with samples from Groups 1 and 2, which were preconditioned, cut to fit 6 ½" X 9 ½" specimen holders, mounted, and placed into the chamber. Using this size of specimen holder allows for nine samples to be exposed to the Fade-ometer at one time. In the first phase of testing, eight samples of three-piece ensembles were exposed to the Carbon Arc Fade-ometer. Four of the three-piece ensembles were from Group 1, which included the outer shell, a Breathe-Tex® moisture barrier, and thermal liner that had not been preconditioned. Four additional samples were from Group 2 which included the outer shell, a moisture barrier, and thermal liner that had not been preconditioned. Four additional samples were from Group 1 which included the outer shell, a moisture barrier, and thermal liner that had not been preconditioned. Four additional samples were from Group 1 which included the outer shell, a moisture barrier, and thermal liner that had not been preconditioned. Four additional samples were from Group 1 which included the outer shell, a moisture barrier, and thermal liner that had not been preconditioned. Four additional samples were from Group 2 which included the outer shell, a moisture barrier, and thermal liner that had been preconditioned. The thermal liner was facing the light source of the Fade-ometer in both Groups 1 and 2. Because only eight three-piece ensemble samples were

exposed in this phase, a ninth specimen holder was available. This ninth specimen holder was filled with two samples of single-moisture barriers that had not been preconditioned. The single-moisture barriers were cut into 4 3/4" X 9 1/2" samples and were stitched horizontally to form a $6 \frac{1}{2}$ " X 9 $\frac{1}{2}$ " sample. The single-moisture barrier sample was placed in the Fade-ometer with the film of the barriers facing the light source. The moisture barriers chosen for this phase of testing were Samples G-Breathe-Tex® and H, both urethane films on an E-89 substrate. The results for Group 1, 2 and 3 are listed in Table 4.1, 4.2, 4.3, and 4.4.

Table 4.1: Phase 1 – Five Hundred Hours of Carbon Arc Fade-ometer Exposure for Group 1

		Microscopic Evaluations			
Hours of Exposure	Hydrostatic Water	Stereo Microscope 7X-35X	Compound Microscope100X		
	Test		Small cell like, yellow and		
Initial	Deeg	Cross with an a oth surface	dark gray, some light pink		
Evaluation	Pass	Gray with smooth surface	areas, lighter in some		
	ĺ		areas, small craters		
20-400*	Pass	Same as initial appearance	Same as above		
420	Pass	Light blue	Same as above		
440-500*	Pass	Same as above	Same as above		

*Examined in 20-hour increments.

		Microscopic Evaluations			
Hours of	Hydrostatic	Stereo Microscope	Compound		
Exposure	Water	7X-35X	Microscope100X		
	Test		Small cell like, yellow and		
Initial	Pass	Gray with crevices giving a	dark gray, some light pink		
Evaluation	1 455	slight wrinkled appearance	areas, lighter in some		
			areas, small craters		
20-400*	Pass	Same as initial appearance	Same as above		
420	Pass	Light blue	Same as above		
440	Pass	Light bluer	Same as above		
460-500*	Pass	Same as above	Same as above		

Table 4.2: Phase 1 – Five Hundred Hours of Carbon Arc Fade-ometer Exposure for Group 2

*Examined in 20-hour increments.

Samples from Groups 1 and 2 produced very similar visual results which was a change in color, as all eight samples changed from gray to a light blue. This color change was not as severe as the color changes seen in the field but was similar to that observed in the field. None of the samples from Groups 1 and 2 failed the Hydrostatic Water Penetration Resistance Test during the 500 hours of exposure in Phase 1. Although microscopic craters were apparent during the initial evaluation of the moisture barrier, the craters did not cause failure.

Table 4.3: Phase 1- Five Hundred Hours of Carbon Arc Fade-ometer Exposure for Single Moisture Barrier-G-Breathe-Tex®

		Microscopic Evaluations			
Hours of	Hydrostatic	Stereo Microscope	Compound		
Exposure	Water	7X-35X	Microscope100X		
	Test		Small cell like, yellow and		
Initial	Pass	Gray with crevices giving a	dark gray, some light pink		
Evaluation	1 ass	wrinkled appearance	areas, lighter in some		
			areas, some small craters		
20	Pass	Lighter gray	Same as above		
40	Pass	Lightening of the gray	Same as above		
60	Pass	Light blue	Same as above		
80	Failed	Same as above	Same as above		

		Microscopic Evaluations			
Hours of Exposure	Hydrostatic Water	Stereo Microscope 7X-35X	Compound Microscope100X		
Initial Evaluation	Test Pass	Yellow/White, areas that look like forming craters	Yellow/slight orange, grainy, craters, lighter areas		
20	Pass	Light Yellow	Same as above		
40	Pass	More yellowing	Same as above		
60	Pass	Darker in color	Same as above		
80-100*	Pass	Same as above	Same as above		
120	Pass	Same as above	Forming cracks		
140-200*	Pass	Same a above	Same as above		
220	Pass	Same as above	Orange/slight pink		
240	Pass	Same as above	More cracking		
260-360*	Pass	Same as above	Same as above		
380	Pass	Dark brown	Same as above		
400-420*	Failed	Same as above	Same as above		

Table 4.4: Phase 1 – Five Hundred Hours of Carbon Arc Fade-ometer Exposure for Single Moisture Barrier-H

*Examined in 20-hour increments.

After five hundred hours of exposure to light in the Carbon Arc Fade-ometer, the only samples that failed the Hydrostatic Water Penetration Resistance Test were single moisture barriers, Samples G-Breathe-Tex® and H. Sample G-Breathe-Tex® failed after 80 hours of exposure. Sample H did not fail until 420 hours of exposure. However, it is important to note that these single-moisture barriers were directly exposed to the Carbon Arc Fade-ometer.

After 60 hours of exposure Sample G appeared visually similar to those changes seen in the moisture barrier of the three-piece ensembles, that is the color changed from gray to light blue, but the moisture barrier in the three-piece ensemble did not fail the water penetration resistance test. The visual changes that occurred in Sample H showed a progression from a yellow/white color to a moderate brown. The results of the singlemoisture barrier samples demonstrate that direct exposure to ultraviolet light causes degradation of the film to occur more rapidly than exposing the moisture barrier as a three-piece ensemble.

Phase 2

In Phase 1 of this study, after five hundred hours of exposure to light in the Carbon Arc Fade-ometer, the only failures that occurred in the moisture barriers were those in a single layer. Therefore, due to available space in the instrument and exposure time to failure, a decision was made to replace all three-piece ensembles except two, one from Group 1 and one from Group 2, with single layer moisture barrier samples. Seven spaces in the Fade-ometer were filled with preconditioned single-moisture barriers identified as Group 3. This included moisture barriers E to M.

Flexing as a pretreatment condition was conducted on Group 3 samples to simulate actual use of the garment and to determine whether flexing would accelerate failure by comparing the results of those samples to the un-flexed three-piece ensembles. However, flexing was not a pretreatment to the three-piece ensembles.

All samples were evaluated for failure after each 20-hour increment of exposure both visually and by using the Hydrostatic Water Penetration Resistance Test. If samples passed the test, that is water did not penetrate through the moisture barrier, they were exposed another 20 hours. This process continued after every 20 hours of exposure. Once failure occurred, the samples were pulled and the number of hours of exposure to failure was recorded. This process was repeated until four replications of a singlemoisture barrier were exposed, the hours of exposure for the samples were averaged and standard deviation calculated. After all single moisture barriers were replicated four times and specimen holders became available in the chamber, one sample from Group 1

and another sample from Group 2 were added to the Carbon Arc Fade-ometer to observe a second replication within these groups. The results will be discussed according to groups of moisture barrier samples.

<u>Group 1.</u> One sample, A, from Phase 1 was continued in Phase 2. This threepiece ensemble sample had been exposed to light for 500 hours without failing the Hydrostatic Water Penetration Resistance Test. Sample C, a three-piece ensemble was added, but this sample was subjected to a flexing pretreatment prior to each 20-hour increment of exposure. The moisture barrier layer, of the three-piece ensemble, was evaluated for water penetration resistance using a Hydrostatic Water Penetration Resistance Test. The number of hours of exposure required to produce failure of the Hydrostatic Water Penetration Resistance Test are listed in Table 4.5.

The moisture barriers of sample C failed the Hydrostatic Water Penetration at 1040 and 1300+ hours of exposure, which was much longer than a single moisture barrier. The moisture barriers of the three-piece ensembles required longer exposure time due to the protection of the film afforded by the thermal liner. Due to the length of time required to produce a leakage failure in the three-piece ensemble and the limitation of only nine specimen holders in the Carbon Arc Fade-ometer, only one replication of each three-piece ensemble was tested.

	Number of Hours of Exposure Before Failure			
Sample	Replication	Average	Standard Deviation	
	1	Average		
Α	1040	1170	183.9	
С	1300+ (sample did not fail)		103.9	

Table 4.5: Phase 2 Summary of Exposure Time to Failure for Group 1

Visually the moisture barriers of Samples A and C, showed a gradual change in color, however, it took several hundred hours before an obvious color change was detected. After five hundred hours of exposure, both samples from Group 1 changed to a light blue color from their original gray. As the exposure time increased and the moisture barrier approached failure the samples changed from a blue/green color to dark blue. The gradual change in color was the only change that occurred in the moisture barriers of the three-piece ensemble and there were no other signs to allow for a prediction of the failure such as cracks or craters. The microscopic evaluations of Group 1 can be found in Appendix A, Tables 1 and 3.

When comparing moisture barrier samples from Group 1 to the garments from the field failures, the color changes in the moisture barrier were not the same, that is samples from Group 1 changed from gray to a shade of dark blue. The color change observed in the field was a shade of blue/green. However, Group 1 samples were not preconditioned, which simulates the cleaning of turnout gear conducted by fire fighters.

The gradual color change of the moisture barrier was due to the protection provided by the thermal liner. However, color change and degradation to the face cloth fabric portion of the quilted thermal liner were detected very early in the exposure. The thermal liners in both the flexed and un-washed three-piece ensembles' produced similar results in that degradation was extreme in both samples.

<u>Group 2.</u> Washing and drying, as per the NFPA 1971 requirements preconditioned all samples in Group 2. For this exposure are moisture barriers were Breathe-Tex®. Sample B, a three-piece ensemble, had been exposed 500 hours in the Carbon Arc Fade-ometer during Phase 1. A second sample, D was added to determine

whether flexing would play a role in the degradation of the moisture barrier. The flexing treatment was conducted prior to each 20-hour increment of exposure to the Carbon Arc Fade-ometer and samples were tested after the treatment for leakage using the Hydrostatic Water Penetration Resistance Test. The number of hours of exposure before failure was determined by the number of hours of exposure required for a sample to fail the Hydrostatic Water Penetration Resistance Test. The results for exposure time for Group 2 are listed in Table 4.6.

	Number of Hours of Ex			
Sample	Replication	Average	Standard	
	1	Average	Deviation	
В	1220	1120	107.2	
D	1040	1130	127.3	

 Table 4.6: Phase 2 Summary of Exposure Time to Failure for Group 2

The exposure of samples from Group 2 failed on an average of 1130 hours of exposure. Sample D failed 180 hours sooner than Sample B. The only difference between Sample B and D was the flexing treatment. Visually, moisture barriers of Samples B and D showed very similar changes. The flexed three-piece ensembles began to show signs of wear, such as wrinkle marks to the moisture barrier and shredding of the thermal liner, which did not affect the moisture barrier's performance. The thermal liner face cloth of these samples changed in color from a dark blue to a brown. The substrates of all three-piece ensembles showed a slight color change.

Samples in Group 2 were previously exposed for 500 hours and only slight color changes were apparent. A summary of the microscopic evaluation before 500 hours can be found in Table 4.2. After 500 hours of exposure until failure, Group 2 samples demonstrated shade changes as being the only significant visual change. Both samples

changed to a lighter gray or a shade of green and continued to darken until failure occurred. No other change was detected in the samples before failure occurred. A summary of the microscopic evaluations of these samples can be found in Appendix A, Tables 2 and 4.

The samples from Group 2 produced similar results to those garments examined from field failures. Group 2 samples, after exposure failure, were a shade of blue/green, which was similar to the color observed in the field garments. The cracking and flaking of the moisture barrier film was not observed in these samples, but failure did occur.

<u>Group 3.</u> This group consisted of samples E-M, which were preconditioned single-moisture barriers. Preconditioned samples were washed and dried and exposed to heat according to NFPA 1971 requirements. The samples were also subjected to the flexing treatment prior to each 20-hour increment of Fade-ometer exposure. Samples were evaluated visually and for water penetration after each 20-hour increment of exposure. Failed samples were removed and replaced with a new sample to allow the testing of another replication. Four replications of each moisture barrier were evaluated, and an average time for exposure calculated. The average and standard deviations for Group 3 are listed in Table 4.7.

		Numbe	er of Hours	s of Exposi	re Before Fai	lure
Sample		Replication				Standard
	1	2	3	4	Average	Deviation
E	200	180	200	180	190.0	11.5
F	160	180	200	200	185.0	19.1
G	100	100	100	100	100.0	0.0
Н	160	200	240	240	210.0	38.3
Ι	140	180	100	140	140.0	32.7
J	20	40	40	40	35.0	10.0
K	20	20	20	20	20.00	0.0
L	100	60	80	100	85.00	19.1
M	180	160	140	140	155.00	19.1

Table 4.7: Phase 2 Summary of Exposure Time to Failure for Group 3

Overall a single moisture barriers required a range of 20-240 hours of exposure before failing the Hydrostatic Water Penetration Resistance Test. The time to failure was much shorter in the exposed single moisture barriers. The moisture barriers from this group required 200 hours of exposure before failure but the only single-moisture barrier sample which exceeded 200 hours of exposure before failure, was Sample H.

When viewed microscopically, changes in appearance were seen in all single moisture barriers before they failed the water penetration test. The changes were different for each single moisture barrier exposed in that some samples showed significant changes while others showed only slight changes.

Sample E – A PTFE film on a pajama check substrate was exposed for an average of 190 hours before failure occurred. When specimens were evaluated visually, the color of Sample E became lighter and continued to lighten until the color began to change to yellow at 140 hours of exposure. Under the compound microscope, fibers from the substrate could be seen through the film after 120 hours of exposure. The results of Carbon Arc Exposure for Sample E are listed in Appendix B, Table 17.

Sample F – A PTFE film on an E-89 substrate, was exposed to the carbon arc for an average of 185 hours before failure occurred. Visually, the color changed to a darker yellow or progressed from a tan to a brown in color from the original white film. As the color of the sample darkened, the area where the Hydrostatic Water Penetration Resistance Testing was lighter than the sample. When examined under the compound microscope, fibers from the substrate could be seen through the film. The results of Carbon Arc Exposure for Sample F are listed in Appendix B, Table 18.

Sample G-Breathe-Tex® – One of the most consistent samples for failure to exposure time was Sample G, which was a urethane film on an E-89 substrate. Each replication of Sample G failed at 100 hours of exposure and therefore, the average exposure time was 100 hours. The film portion of replications 1, 2, and 3 of Sample G progressively changed from a gray to a light blue before turning white. Replication 4 went from a darker gray to a lighter gray with a yellow cast. When viewed under the compound microscope, cracks were obvious between 80 and 100 hours of exposure. The results of Carbon Arc Exposure for Sample G are listed in Appendix B, Table 19.

Sample H – Sample H, a urethane film on an E-89 substrate, required the longest period of exposure to reach failure of all the samples, that is an average of 210 hours. Like Sample G, the film of Sample H cracked before failure in all four of the replications. Visual color changes showed that the samples progressively lightened except for Replication 2, which appeared darker in color in the exposed area only. The results of Carbon Arc Exposure for Sample H are listed in Appendix B, Table 20.

Sample I – Was constructed of a neoprene coating on a polyester and cotton substrate. This moisture barrier sample was exposed an average of 140 hours before

failing the Hydrostatic Water Penetration Resistance Test. Visually, Sample I was very different from the other moisture barrier samples. Instead of getting lighter or turning yellow, the color of Sample I changed from white to a very dark brown. Cracks could be seen in all replications after 40 hours of exposure, but failure did not occur in this sample until between 100 and 180 hours of exposure were completed. The cracking progressively worsened before failure occurred. Also, the sample became very brittle to touch and produced a scorched smell for all replications after 20 hours of exposure. Wrinkled marks were apparent after flexing and the samples were lighter in color in the area of the hydrostatic test. The results of Carbon Arc Exposure for Sample I are listed in Appendix B, Table 21.

Sample J – Sample J, a urethane film on an E-89 substrate, required one of the lowest numbers of hours of exposure before failure occurred, with an average exposure time of 35.0 hours. The color of all replications of Sample J progressed from a yellow to a dark yellow and cracks or other visual changes were seen under the compound microscope. The results of Carbon Arc Exposure for Sample J are listed in Appendix B, Table 22.

Sample K – A urethane film on an E-89 substrate was not only one of the most consistent in replicating the number of exposure times before failure, but also required the lowest exposure times. Sample K failed at 20 hours of exposure in all four replications. Sample K, like Sample J, changed color from a light yellow to a dark yellow. The results of Carbon Arc Exposure for Sample K are listed in Appendix B, Table 23.

Sample L – A PTFE film on a Vilene substrate, took an average of 85 hours of exposure before failure occurred. Visually, the film in all four replications of this sample consistently changed colors going from a white to a pink. A small crater appeared on the film of Replication 1 after 40 hours of exposure. However, failure of this replication did not occur until after 100 hours of exposure. The results of Carbon Arc Exposure for Sample L are listed in Appendix B, Table 24.

Sample M – This sample was a urethane film on a Vilene substrate, which failed after an average of 155 hours of exposure. Visually, Sample M turned from yellow/white to a dark yellow or tan. Also, craters were visible for replications 1 and 2 after 40 hours of exposure. The results of Carbon Arc Exposure for Sample M are listed in Appendix B, Table 25.

To summarize the results of Groups 1, 2, and 3 of Phase 2, those moisture barriers samples that demonstrated very little change in color also required more hours of exposure before failure. Those that showed similar changes during each replication also required similar hours of exposure before failure within the replication. The samples that experienced significant changes within the first 40 hours of exposure failed sooner than those that went through a slow progression of color change or surface integrity.

As was apparent from the high standard deviations, the variability between exposure times to failure was large for some samples. Samples were only evaluated in 20-hour increments; therefore when a sample reached failure it was reported as failing at that 20-hour increment. Due to the 20 hour time span between each assessment, it could not be determined if the failure occurred in the first several hours of the 20 hour exposure or at the end of the 20 hour exposure. If samples could have been evaluated every hour,

the averages and standard deviations would have been different, possibly providing a lower standard deviation. The averages and standard deviations for Group 3 samples are listed in Table 4.7.

When comparing the moisture barriers from field garments to Group 3 of Carbon Arc exposure only Sample G moisture barriers were examined, because it was the only Breathe-Tex® sample included in Group 3. After exposure Sample G changed colors from a gray to a white before failing the Hydrostatic Water Penetration Resistance Test. Even though color change occurred it was not the same blue/green seen in field failure. However, this sample was exposed as a single-moisture barrier and not as a three-piece ensemble. Cracking of the film in Sample G moisture barrier was apparent after exposure to the Carbon Arc Fade-ometer. The cracks were similar to the cracking of the film in field failures. Other samples in Group 3 were not evaluated as field garments, but all samples in Group 3 did demonstrate color change and failure to the Hydrostatic Water Penetration Resistance Test.

Natural Sunlight Exposure

Phase 3

Phase 3 testing was conducted to determine if natural sunlight exposure affected the degradation of the moisture barrier and to enable a comparison between the results from natural sunlight to an artificial ultraviolet light, i.e., the Carbon Arc Fade-ometer. This phase of testing involved exposing 252 moisture barriers to natural sunlight at a Q-Panel Lab facility in Florida. For purposes of reporting and discussing the results, moisture barriers were grouped using the same sample identification as those exposed to instrumental light in the Carbon arc. Group 2 samples were three-piece ensembles,

which were preconditioned prior to exposure according to NFPA 1971 Standard 6-1.2 (2000 Edition) but flexing was not a pretreatment for the moisture barriers. Half of the samples from Group 2 were exposed with the thermal liner facing the light source while the other half exposed the outer shell to the natural sunlight. Group 3 samples were single-moisture barriers that were also preconditioned according to NFPA 1971 Standard 6-1.2 (2000 Edition) but also were not flexed. During exposure to natural sunlight, the film side of the moisture barrier was face up to provide the greatest opportunity for exposure to sunlight. Fourteen specimens per moisture barrier type were replicated for each sample in Groups 2 and 3. Each week, one specimen from each type of moisture barrier was pulled and its performance was evaluated for water penetration using the Hydrostatic Water Penetration Resistance Test and visually using the stereo and compound microscopes.

Because of the cost of exposure and the time required for shipping, samples could not be returned for further exposure if they passed the water penetration test. Hence, there were no duplicate replications of the single layer moisture barrier types in the natural sunlight exposure. However, when the first failure occurred in a sample, the failure continued in the following weeks of exposure for the same moisture barrier and the data was reported as the number of weeks of sunlight exposure required before failure occurred. The results for natural sunlight exposure time for Groups 2 and 3 are listed in Table 4.8 and Table 4.9.

Sample	Fourteen Weeks of Exposure Results
В	Did not fail
Ν	Did not fail
0	Did not fail
Р	Did not fail
Q	Did not fail
R	Did not fail
S	Did not fail
Т	Did not fail
U	Did not fail
V	Did not fail
W	Did not fail
X	Did not fail

Table 4.8: Group 2 Results for 14 Weeks of Natural Sunlight Exposure

<u>Group 2.</u> All of the moisture barriers included in this group of three-piece ensembles passed the Hydrostatic Water Penetration Resistance Test during the entire fourteen-week period of sunlight exposure. After 14 weeks of exposure to natural sunlight the changes in the moisture barriers were visual changes. Even the visual changes only involved a change in color and there were no apparent structural changes to any of the moisture barriers from this group. A discussion of the results will be presented by individual sample or by grouping those moisture barrier samples with the same composition but differing in their orientation during exposure.

Sample B and N – These samples were three-piece ensemble samples, with Breathe-Tex® a urethane film on an E-89 substrate moisture barrier. They were exposed with the thermal liner facing the light source for Sample B and the outer shell facing the light source for Sample N. The first noticeable change in these samples was a color change, from a yellow and dark gray to a dark red and a progression to a blue/gray and then to blue as it was exposed from 1 to 5 weeks of sunlight. This blue color darkened and brightened through the remaining weeks of exposure. The results of Natural Sunlight

Exposure for Sample B are listed in Appendix A, Table 5 and Sample N in Appendix A, Table 6.

Samples 0, P and W – These three samples are composed of PTFE films on pajama check substrates. Color changes of these samples, which were originally white, were viewed as brown which progressively darkened during Weeks 11 through 13. No additional changes occurred in the remaining weeks of exposure. The results of Natural Sunlight Exposure for Sample O are listed in Appendix A, Table 7, Sample P in Appendix A, Table 8, and Sample W in Appendix A, Table 15.

Samples Q and R – Both samples were a urethane film on E-89 substrates, with Sample Q's outer shell facing the light source and Sample R's thermal liner facing the light source. The change in both of these moisture barriers was a visual change in color from a white film to a yellow which darkened with additional exposure and was bright yellow at the end of 10 weeks. The results of Natural Sunlight Exposure for Sample Q are listed in Appendix A, Table 9, and Sample R in Appendix A, Table 10.

Samples S and T – Samples S and T, were both PTFE films on a Vilene substrate. During exposure Sample S had the outer shell facing the light source and Sample T had the thermal liner facing the light source. Whether the shell or the thermal liner was facing the light, the only change in the moisture barrier was a visual one from a white to a light pink after eight weeks of exposure, which progressed to a light brown shade after the ninth week. The results of Natural Sunlight Exposure for Sample S are listed in Appendix A, Table 11 and Sample T in Appendix A, Table 12.

Sample U and V – These samples, urethane films of Vilene substrates, were of the same fabric composition, except that during exposure Sample U's outer shell faced the

light source, and Sample V's thermal liner was facing the light source. After fourteen weeks of exposure, the results were the same for both samples, but the changes progressed faster for Sample V than Sample U. Originally, both samples were a yellowish white in color that changed to a moderate brown by the fourteenth week of exposure. After the first week of exposure these samples became brittle and chalky. No other changes were seen for both samples. The results of Natural Sunlight Exposure for Sample U are listed in Appendix A, Table 13 and Sample V in Appendix A, Table 14.

Sample X – This sample was very similar to Samples S and T, in that it was a PTFE film on a Vilene substrate, however the sample composite was constructed using a different type of thermal liner called GlideTMPure. Although the thermal liner was different, the end results were similar to those observed for Samples S and T. The color of the sample progressively changed from a white film to medium brown film by Week 13. There were no other apparent changes in this sample. The results of Natural Sunlight Exposure for Sample X are listed in Appendix A, Table 16.

In comparing the results of natural sunlight exposure to the moisture barriers within the failures from the field, only Samples B and N were of the same type. All other samples in Group 2 of natural sunlight exposure were constructed of different moisture barriers than the field garments. Although Samples B and N did not fail the water penetration test after fourteen weeks of exposure, they did show similar visual results as the moisture barriers of the failed garments from the field. The same color change occurred and the appearance of craters was obvious.

Sample	Fourteen Weeks of Exposure Results
E	Did not fail
F	Failed at Week 5 only
G	Failed at Week 3 through Week14
L	Failed at Week 12 and Week 13
M	Failed at Week 6 through Week 14, Except Week 8
Y	Failed at Week 2 through Week 14

Table 4.9: Group 3 Results for 14 Weeks of Natural Sunlight Exposure

<u>Group3.</u> Samples in Group 3 moisture barriers were constructed of different components. Group 3 samples that had been exposed to instrumental light exposure in the Carbon-Arc were also exposed to natural sunlight, these are Samples E, F, G, L and M. Sample Y was added to the natural sunlight exposure but was not included in the instrumental light treatment

Sample E – Which was a PTFE film on a pajama check substrate, did not fail the water penetration test during the entire treatment to sunlight exposure. However, the appearance changed, as the color of the film changed from an orange/yellow to a bright yellow with a slight orange cast, which progressively darkened to a brown before the test was terminated after fourteen weeks. The results of Natural Sunlight Exposure for Sample E are listed in Appendix B, Table 26.

Sample F – A PTFE film on an E-89 substrate had a specimen fail the Hydrostatic Water Penetration Test after five weeks of exposure. But, the failure was not replicated during the remaining fourteen weeks of sunlight exposure. A reason for the failure of the one specimen was not determined, however the moisture barrier could have had a thin or flawed area which caused the failure. Otherwise, the changes in Sample F were very similar to those of Sample E. Sample F, a white film with the substrate visible through the film. The color of the film changed to a light yellow with a dark orange substrate

after four weeks, and with continuous exposure the substrate turned brown.

Delamination, separation of the film from the substrate, was apparent after ten weeks of exposure, which could indicate that sunlight exposure had degraded the adhesive used to laminate the film to the substrate. The results of Natural Sunlight Exposure for Sample F are listed in Appendix B, Table 27.

Sample G-Breathe-Tex® – This sample, a urethane film on an E-89 substrate, was the second moisture barrier to fail the water penetration test during the natural sunlight treatment. Failure occurred after the third week and continued throughout the remaining weeks of exposure. Visually, significant changes began to occur after the first week of exposure, in that the sample changed from a gray to a yellow. This color changed progressed after each week of exposure until the sample was dark yellow after being exposed for fourteen consecutive weeks. After three weeks of sunlight exposure, Sample G's film showed cracking and the grainy appearance initially viewed in this sample was darkened and became more visible. Cracking increased and flaking away of the film began as the film became very brittle. The results of Natural Sunlight Exposure for Sample G are listed in Appendix B, Table 28.

Sample L – A PTFE film on a Vilene substrate failed the water penetration test after twelve weeks of exposure to the natural sunlight environment. Visual changes in the color were apparent after the first week of exposure, as it changed from yellow to a light brown color. After four weeks of exposure the color changed to pink and, with additional exposure, to a medium brown, which became apparent after eleven weeks of exposure. The results of Natural Sunlight Exposure for Sample L are listed in Appendix B, Table 29.

Sample M – Sample M, a urethane film on a Vilene substrate, failed after six weeks of exposure to natural sunlight and continued to fail in all remaining weeks except for Week 8. The visual changes to Sample M were a gradual progression from a dull shade of yellow to a bright yellow and the appearance of cracks and craters. This sample changed from a shiny film to a brittle and chalky film after exposure. After eleven weeks of exposure the cracks in the film increased and the color changed to an orange. The results of Natural Sunlight Exposure for Sample M are listed in Appendix B, Table 30.

Sample Y – Sample Y is very similar to Sample G, being a urethane film on an E-89 substrate, although Sample G was initially gray and Sample Y was white. Sample Y failed after two weeks of exposure and Sample G failed after three weeks of exposure. After one week of exposure, Sample Y's color changed from white to yellow with a dark grainy texture, when viewed under the microscope. After two weeks of exposure, the sample had become a darker yellow in appearance and cracking was apparent under the compound microscope. In the remaining weeks of exposure, more cracking and flaking of the film was apparent. The results of Natural Sunlight Exposure for Sample Y are listed in Appendix B, Table 31.

When comparing the results of natural sunlight exposure to the moisture barriers that have experienced failures in the field, some of the moisture barriers did allow water penetration and experienced change in color and/or cracking and flaking of the films during the 14 weeks of exposure to natural sunlight. However, not all of the moisture barriers failed the Hydrostatic Water Penetration even after 14 weeks of natural sunlight exposure. For example, none of samples from Group 2 failed during the entire 14 weeks of exposure. But the only moisture barrier samples that were of the same construction as

the moisture barriers that failed in the field were Samples B and N. Although these two samples did not fail the water penetration test after fourteen weeks of exposure, they did show the same color change as the moisture barriers of the garments from the field.

In Group 3 moisture barriers, the only moisture barrier type that duplicated Breathe-Tex®, the moisture barrier that had failed in the field, were Samples G and Y. When subjected to sunlight exposure, the two samples exhibited a change in color but the colors were not the same as the moisture barriers evaluated from the field garments. The original gray color of both samples changed to yellow, which progressively darkened with continuous exposure. In contrast, both G and Y experienced severe cracking and flaking of the film prior to failure, which was similar to the degradation observed in garments from the field. Other moisture barrier samples from Group 3 also showed cracking and flaking of the films prior to failing the water penetration test. Since failure occurred to these other moisture barriers exposed to natural sunlight, failure would also occur in the field.

Hydrostatic Water Penetration Resistance Test

Hydrostatic Water Penetration Resistance Test was used as a performance measure to test the resistance of the moisture barriers to water penetration. The test was initially used to prescreen the moisture barriers prior to exposure in the Carbon Arc Fadeometer and the sample was discarded if it failed the test. During the process of prescreening the specimens for the instrumental light treatment only two such failures occurred. Therefore, the assessment for water penetration was applied randomly to 20% of the moisture barriers used in the natural sunlight treatment.

After 20-hour increments of instrumental light exposure with and without flexing of the samples, the Hydrostatic Test was the performance measure that determined whether the product failed and was removed from the study or passed and the exposure continued. The presentation and discussion of the results will be according to the type of treatment applied to the moisture barriers.

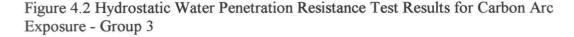
Carbon Arc Fade-ometer Exposure

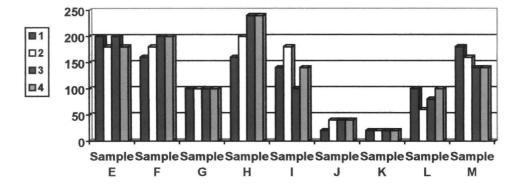
<u>Groups 1 and 2.</u> Breathe-Tex® moisture barriers assembled as part of a threepiece ensemble, exposed through the thermal liner to the Carbon Arc Fade-ometer, did not fail the Hydrostatic Water Penetration Resistance Test until after 1000 hours of exposure. Although craters and/or thinning of the film were apparent during the initial microscopic evaluation of samples this did not cause leakage to occur as Sample A did not fail until 1040 hours of exposure and Sample B's failure occurred after 1220 hours of exposure. Sample C had not failed when the treatment stopped at 1300 hours of exposure and sample D failed after 1040 hours of exposure. The Hydrostatic Water Penetration Resistance Test results and microscopic evaluations for Carbon Arc exposure for Group 1, Samples A,B,C, and D are listed in Appendix A, Tables 1,2, and 3.

<u>Group 3.</u> Samples were exposed to the light source as single layer moisture barriers. The performance results of the Water Penetration Resistance Test exhibited much greater variability from sample to sample. In the initial evaluation of these samples, craters were observed, as well as large thinned areas on the film, which allowed light to pass through. The difference in the moisture barriers exposed as a single layer was that some samples had cracks or areas of film delamination from the substrate after exposure to the Carbon Arc Fade-ometer. The results of the Hydrostatic Water

Penetration Resistance Test to Carbon Arc exposure for Group 3 are presented in Figure

4.1 and detailed descriptions of the results are presented in Appendix B.





In the initial evaluation of Samples E and F, craters were apparent in the film layer, but the samples passed the Hydrostatic Water Penetration Resistance Test and leakage did not occur until more than 185 hours of exposure. Craters were also observed during the initial screening of Sample G, H, I but the craters did not result in failure. At the time of failure all replications for Sample G, H, and I showed cracking of the film side of the moisture barrier. The appearance of cracking did not always predict failure, for example in some samples cracking appeared but additional exposure time was needed before the sample failed the water penetration test. One could assume that cracks were beginning to form but they were not deep enough to allow water to penetrate the film. As exposure continued, the cracks became longer and deeper and/or more cracking occurred.

Samples J, K, L and M moisture barriers generally did not show the presence of craters during the initial visual evaluations, except for one replication of Sample L in which one small crater was present. Moisture barrier M developed some craters after 40 hours of exposure. Samples J, K, L and M did not exhibit the presence of cracking of the films at the time of failure to the Hydrostatic Water penetration test.

Natural Sunlight Exposure

Group 2. Consisted of moisture barriers exposed to natural sunlight as part of a three-piece ensemble. All samples in this group passed the Hydrostatic Water Penetration test throughout the entire length of the exposure. However, the observations of craters were comparable to the samples exposed to instrumental light. In Samples B and N, Breathe-Tex® moisture barriers, craters were apparent during the initial evaluation and in Samples O, P and W *c*raters were observed but did not contribute to leakage of the moisture barriers.

Samples Q, R, S, U, V and X moisture barriers were all samples in which craters were not visible during the initial screening nor throughout the fourteen weeks of exposure. Sample T did show craters after Weeks 8 and 9 but failure did not occur.

Group 3. In the initial evaluation of Sample E, craters were apparent in the film layer but the samples passed the Hydrostatic Water Penetration Resistance Test and leakage did not occur throughout the natural sunlight exposure. Craters were also observed during the initial screening of Sample G and Y, which are both Breathe-Tex®, but the craters did not result in failure. After the first week of exposure, cracking of the film side of the moisture barriers was obvious. For both samples, cracking was apparent before failure occurred but additional sunlight exposure was needed before the sample failed the water penetration test. One could assume that cracks were beginning to form but the crack was not deep enough to allow water to penetrate through the film. As exposure continued the cracks became longer and deeper and/or more cracking occurred.

Initially moisture barrier Samples F and L did not show the presence of craters, however the film layer of these samples delaminated after ten weeks of sunlight

exposure. When delamination first occurred both samples passed the Hydrostatic Water Penetration Test, which indicates that the delaminated film remained in tact for two weeks because Sample L did fail the water penetration test after 12 weeks of exposure.

Sample M moisture barrier did not exhibit the presence of craters during the initial visual evaluations, however cracking of the film layer was apparent after four weeks of sunlight exposure. The same cracking trend observed in Samples G and Y also occurred in Sample M. When cracking first occurred the sample passed the Hydrostatic Water Penetration Test, but failure did occur after additional weeks of exposure.

In summary, craters and crevices which were apparent in the initial visual examination and/or continued throughout the treatments, did not predict potential failure of the Hydrostatic Water Penetration Resistance Test. Craters are tiny thinned spots of the film where light passes through and crevices are thinned sections of the film that allow lines of light or larger areas in which light passes through the film during microscopic examinations. In contrast, cracking and/or delamination of the film were both visual indicators that leakage would occur after the treatment of light exposure. In some samples cracking and delamination were apparent and leakage did not occur but as the degradation increased the moisture barriers always failed the Water Penetration Test.

Comparison of Instrumental Light and Natural Sunlight to Field Failures

Of the hundreds of garments where failure was seen in the field, five were selected to conduct a closer examination, to establish a baseline for comparison to instrumental and natural sunlight exposures. The 5 garments were selected because of their representation of the type of degradation seen in the field. The color of the moisture barriers in all 5 garments had changed from a gray to a blue/green color. The film layer

had also degraded as there was evidence of severe cracking and flaking of the film and sections of the film were missing and these moisture barriers obviously failed the Hydrostatic Water Penetration Resistance Test.

After instrumental exposure to the Carbon Arc Fade-ometer, moisture barriers from the three-piece ensembles in Group 1 did experience a change in color with a progression from gray to dark blue. The color change was not the same as seen in the field, however these samples were not preconditioned. Preconditioned samples could more closely simulate washing and wear of field garments.

After exposure to the Carbon Arc Fade-ometer, moisture barriers from the threepiece ensembles in Group 2, showed similar visual changes to the Breathe-Tex® moisture barriers examined in the field. The color of these moisture barriers were originally gray but changed to a blue/green, which was very comparable to the color observed in the garments from the field. In both Groups 1 and 2, cracking and flaking, which was observed in the field garments, was not apparent, however leakage failure did occur in all but one sample from Group 1.

After natural sunlight exposure, even though none of the moisture barriers exposed as a three-piece ensemble in Group 2 failed the water penetration resistance test, similar visual changes were apparent for all samples. The color change and craters were apparent. Only two of the moisture barrier samples in the group were Breathe-Tex®, the moisture barriers found in the failures in field.

Group 3 moisture barrier samples did show visual changes and failed the Hydrostatic Water Penetration Resistance Test after exposure to instrumental light in the Carbon Arc Fade-ometer. Even though only one sample from this group was of the same

type of moisture barrier as the type of moisture barrier in the field garments, the other samples showed similar visual changes and failed the water penetration resistance test. The color change in Group 3 moisture barriers was not the same change in color observed in garments from the field, however cracking was apparent after exposure, which was the same type of degradation seen in field garments.

When exposed to natural sunlight, Group 3 moisture barriers also produced visual changes that were similar to the moisture barriers examined from field failures. The changes were primarily in color, cracks or delamination of the moisture barrier's film or coating. In contrast to the instrumental light exposure, not all moisture barriers exposed to natural sunlight failed the water penetration resistance test even though there were obvious changes in the product. However, only two samples in Group 3 moisture barriers were the same type of moisture barrier as those observed in the failed garments from the field. These two samples did not show the same change in color but cracking and flaking of the film was apparent before they failed the Hydrostatic Water Penetration Resistance Test. One could assume that failure would occur after further exposure based on the similarities in the changes of appearance.

Research Questions

To answer the research questions developed for this study, the results were compared and statistical analysis of the data was conducted. Results of the two light exposures and flexing were compared to determine whether failure in the moisture barrier was caused by ultraviolet light exposure. A comparison of the flexed and un-flexed samples was conducted to determine if failure in the moisture barrier was affected by abrasion. Finally, statistical analysis of the data was conducted to determine if damage to

the moisture barrier could be replicated to develop a test method that would predict failure.

Research Question # 1. Is the failure in moisture barriers caused by ultraviolet light exposure?

Instrumental and natural sunlight exposures were used in this study to enable the researcher to determine if the degradation of the moisture barrier layer of the fire fighter's turnout gear was caused by ultraviolet light. The exposure to instrumental light was conducted in the Carbon Arc Fade-ometer, which transmits light in the 275-370 nanometer range and ultraviolet light, which is 250-400 nanometers. The light source of the Carbon Arc Fade-ometer transmits light in the mid range of ultraviolet light when compared to natural sunlight, which enables the instrument to accelerate the exposure of a sample. Since instrumental exposures may not duplicate natural sunlight, the moisture barriers were also exposed to natural sunlight. This treatment not only enabled comparison to the instrumental light sources but also exposed the samples to an environment which is similar to that in the field.

When evaluating the results from the Carbon Arc Fade-ometer exposure, all single moisture barriers failed the Hydrostatic Water Penetration Resistance Test, but the length of exposure time to achieve failure varied with the type of moisture barrier. The moisture barriers exposed through the thermal liner as part of a three-piece ensemble in the Carbon Arc also failed the Hydrostatic Water Penetration Resistance Test. In comparing the results of exposing single moisture barriers and the moisture barriers as a three piece ensemble in the Carbon Arc Fade-ometer, single moisture barriers failed

sooner because of direct exposure whereas the three-piece ensembles were protected by other layers that filtered out much of the light.

The results of natural sunlight also showed that failures in the single moisture barriers occurred much sooner than the moisture barriers protected by the three-piece ensemble. As with instrumental light exposure the exact color changes were not duplicated but a change in color was evident and moisture barriers that failed produced cracking and/or delamination of the film.

The multivariate statistical test used to analyze the resulting data from the instrumental and natural sunlight exposure did not show significant results. However, when visually compared to the product failures of field garments the results were very similar. Also, when evaluated for water penetration, samples that failed from instrumental exposure exhibited similar results to reported failures in the field. In the preliminary investigation of the field moisture barriers, failures were more prominent when the thermal liner had been exposed to ultraviolet light. These areas were a greenish blue with cracking and flaking. In instrumental exposure where the thermal liner was exposed to the light source, the same color change was seen before failure occurred. Therefore, the failure seen in the field has been replicated by using instrumental ultraviolet light and the failure may be attributed to filtered ultraviolet light.

Studies conducted by Day et al, concluded that exposure to light reduced the strength of protective clothing fabrics (1988). The thermal liner of the flexed three-piece ensemble was completely degraded following exposure and the single moisture barriers exposed to instrumental and natural sunlight showed degradation of the film, which caused failure to occur.

Research Question # 2. Is the moisture barrier's breakdown affected by abrasion?

Abrasion is the rubbing of one object against another. The rubbing may cause wear to occur to the abraded object's surface. Examples of wear from abrasion is seen as thinned areas, broken fibers, pilling, holes, cracking, weight loss and many other types of degradation. The single moisture barriers used in the Carbon Arc exposure were flexed, which simulates a form of edge and surface abrasion.

The moisture barriers exposed as a three-piece ensemble included one sample in Group 1, Phase 2 flexed prior to exposure to the Carbon Arc Fade-ometer. In Group 2, Phase 2 exposure to instrumental light included one flexed moisture barrier sample and one that was not. The results of this moisture barrier showed that the flexed sample failed 80 hours sooner than the un-flexed sample. Therefore, a decision was made to include flexing as a pretreatment for all moisture barriers in the Carbon Arc Fade-ometer treatment. Hence, a comparison of flexed and unflexed samples did not apply to the moisture barriers in Group 3, Phase 2 because all samples were flexed as an initial pretreatment and prior to each 20- hour increment of exposure.

Vogelpohl's study of used turnout gear found that failure to water penetration could be related to abrasion (1996). Mäkinen suggested when testing the effects of laundering, wear to materials should also be included (1992). The results of this study did not show significant difference in the results when comparing flexed and un-flexed moisture barrier samples. However, results of abrasion tests conducted by Vogelpohl and Mäkinen indicate that abrasion may contribute and can be a cause of failure and material wear to occur.

Research Question # 3. Can the damage be replicated in order to develop a test method that will predict failure?

A problem had been reported with the moisture barrier layer of fire fighter's turnout gear. Five problem garments were evaluated to assess their appearance and performance. Visually the degradation in the moisture barriers of field garments was seen as a definite change in color from an original gray to a greenish-blue with the film layer of the moisture barrier experiencing severe cracking and flaking. In multiple locations the film was thinning or was completely missing from the substrate. The water penetration resistance test was not conducted due to the flaking and loss of film observed of the moisture barriers, which would obviously cause failure to occur.

When comparing the moisture barriers of failed field garments to the instrumental exposure using the Carbon Arc Fade-ometer, not all samples exhibited similar changes. For example, the moisture barriers of three-piece ensembles that were not preconditioned showed visual color changes from the original gray to dark blue. Moisture barriers of three-piece ensembles that were preconditioned showed similar color changes to those of the failed field garments, in that the original gray color appeared blue/green after exposure. Cracking and flaking of the moisture barrier films of three-piece ensembles was not apparent, however failure in the Hydrostatic Water Penetration Resistance Test did occur in all samples except one. The single-moisture barriers exposed to the Carbon Arc Fade-ometer were of a different composition. Although not all samples were the same as the failed field garments, leakage failure did occur in samples. The initial colors and some of the color changes were not the same as the garments in the field but cracking was apparent in some single moisture barriers. One single-moisture barrier sample, of

the same composition, as the field composition did not show similar color changes, however cracking was apparent before leakage failure.

In comparing the field failures to the results of the samples exposed to natural sunlight, degradation was also replicated. None of the samples from Group 2 failed after fourteen weeks of exposure but visual changes similar to those observed in the field garments were seen. Two samples from Group 2 were of the same composition as those failed garments in the field. Although failure and cracking did not occur after fourteen weeks of exposure, the color change was similar to those observed in the field garments. Not all samples from Group 3 failed the Hydrostatic Water Penetration Resistance Test, but other forms of degradation, which was observed in the field garments, were replicated.

The degradation assessed in the field garments was replicated in all exposed three-piece ensembles. Failure only occurred in the Carbon Arc exposed samples but the same color changes were seen in both sunlight exposure and field garments. The degradation seen in the single moisture barriers, which were directly exposed to ultraviolet light occurred after fewer hours of exposure than in the three-piece ensembles.

Chapter Five

Summary and Conclusions

The purpose of this research was to investigate the failures seen in the moisture barrier of fire fighter's turnout gear. In field use, the moisture barrier layer of turnout gear was degrading and no longer providing protection from water and hazardous liquids penetration. The specific objectives of the study were to determine the cause of failure and to develop a test method that replicates the failure for testing of all moisture barrier materials.

In an attempt to solve this problem, garments, which had failed in the field, were examined. The moisture barriers had changed color from an original gray to a blue/green color and severe cracking and flaking of the film was apparent. Cracking and flaking or complete loss of the film layer of the moisture barrier films were so severe that it was obvious that the product would not pass a water penetration resistance test.

The research designed to address this problem included subjecting multiple replications of nine different types of moisture barriers to two exposures. Treatments one and two included exposure to artificial and natural sunlight. The effect of light on the moisture barriers was evaluated visually using stereo and compound microscopes and for performance by testing for water penetration using the Hydrostatic Water Penetration Resistance Test. The third treatment involved flexing the moisture barrier to simulate flexing and surface abrasion.

The first objective of the study was to determine a cause of failure in the moisture barrier of the fire fighter's protective clothing. Results of the study found that after instrumental exposure to the Carbon Arc Fade-ometer, moisture barriers from the three-

piece ensembles did experience a change in color. The samples that had not been preconditioned exhibited a change in color from gray to dark blue, which was not the same color change as seen in the field, however, moisture barriers that were preconditioned showed similar visual changes to those garments in the field after exposure to the Carbon Arc Fade-ometer. The color of these moisture barriers was originally gray but changed to a blue/green, which was very comparable to the color observed in the garments from the field. The cracking and flaking, which was observed in the field garments, was not apparent. However, all but one sample failed the Hydrostatic Water Penetration Resistance Test. The results of a fourteen weeks exposure to natural sunlight showed that even though none of the moisture barriers exposed as part of a three-piece ensemble failed the water penetration resistance test, visual changes similar of those failures in the field, were apparent for all samples.

When moisture barriers were exposed as single layers to both instrumental and natural sunlight, the results were much closer to replicating the damage that had occurred in the field. All moisture barrier samples exposed to instrumental light in the Carbon Arc Fade-ometer failed the Hydrostatic Water Penetration Resistance Test after exposure and exhibited visual changes that were very similar to the failures in the field. The color changed in all moisture barriers and in some moisture barriers cracking and/or delamination of the film or coating was apparent, which was the same type of degradation as seen in field garments.

When exposed to natural sunlight, single moisture barriers also produced visual changes that were similar to the moisture barriers examined from field failures. The changes were primarily color and cracks or delamination of the moisture barrier's films

or coatings. In contrast to the instrumental light exposure, not all moisture barriers exposed to natural sunlight failed the water penetration resistance test, even though there were obvious changes occurring in the product. However, the samples that were the same type of moisture barrier as the garments from the field, failures, showed the same change in color and cracking of the film. Flaking of the film was also apparent before they failed the Hydrostatic Water Penetration Resistance Test.

Therefore, the researcher concludes that ultraviolet light did contribute to failure of the moisture barrier of fire fighter's turnout gear. In the preliminary investigation of the moisture barrier failures from the field, areas where failure was more prominent were sections in which the thermal barrier was exposed to ultraviolet light. The moisture barriers from these areas were a blue/green color that were originally gray and the film showed severe cracking or flaking. In this study, when the three-piece ensemble was positioned with the thermal liner exposed to the light source, in both instrumental and natural sunlight the same color change in the moisture barrier was seen before failure occurred.

When the single moisture barriers were exposed to instrumental and natural sunlight, the visual changes did include color change but the same change from gray to blue/green was not always apparent. Cracking and/or flaking of the film portion of the moisture barrier was apparent and all but one sample failed the Hydrostatic Water Penetration Resistance Test. Therefore, the failure seen in the field has been replicated by using instrumental ultraviolet light and natural sunlight and the failure of the moisture barrier was attributed to filtered ultraviolet light.

The second objective of the study was to develop a test method to replicate the failure for future testing. The research findings identified some of the parameters that should be included in a test method that can be used to evaluate moisture barriers during product development and/or products in use.

Initial examination of the moisture barriers from garments that had failed in the field showed a definite change in color from an original gray to a blue/green and the film layer of the moisture barrier experienced severe cracking and flaking. A performance assessment using the Hydrostatic Water Penetration Resistance Test was not conducted due to the flaking and loss of film observed in these moisture barriers. The results of this study showed that moisture barriers exposed as a single layer to the instrumental exposure using the Carbon Arc Fade-ometer exhibited similar changes. The moisture barriers of the same type as the products from the field failure experience similar color changes. Cracking and flaking of the moisture barrier films was also apparent before the product failed the Hydrostatic Water Penetration Resistance Test. Although not all moisture barrier samples were the same as the failed field garments, leakage failures also occurred when these samples were exposed to the Carbon Arc Fade-ometer. Visual color changes were not the same as the garments in the field but cracking and/or delamination were apparent in some single moisture barriers.

Exposure to natural sunlight also enabled the researcher to replicate field failures. Even though not all samples failed the Hydrostatic Water Penetration Resistance Test, other forms of degradation were observed in the moisture barriers after exposure to natural sunlight. The moisture barriers that were of the same type as those from the

failed garments in the field showed a different color change but cracking and flaking of the film was apparent and they failed the Hydrostatic Water Penetration Resistance Test.

In conclusion, the parameters that should be included in the test method to predict failures of the moisture barrier are preconditioning of the sample, exposure to an ultraviolet light source as a single layer and an abrasion pretreatment. The test method should include at least a visual assessment of color and microscopic examination as well as a performance test for penetration.

This conclusion is based on the results of this study in which all moisture barrier samples that were subjected to the NFPA 1971 Standard 6-1.2 (2000 Edition) pretreatment failed the water penetration resistance test after exposure to both instrumental and natural light. A test method should include exposure to light as a treatment for the moisture barriers. This is based on the results of this study in which both instrumental and natural sunlight were able to reproduce the type of failures seen in the field. Light exposure testing is both time-consuming and expensive to conduct. Therefore, the researcher suggests that single layer moisture barriers should be used for testing, at least in the development of a new product. This conclusion was supported by the findings, which showed that failure occurred much sooner when the moisture barrier was exposed as a single layer.

The parameter of light source merits further investigation before a test method can be developed. The results of this investigation showed that both instrumental and natural sunlight produced visual results similar to those seen in failed garments from the field. The time required to test a sample would be a major consideration. For example, the evaluation of the moisture barrier that was the same type as those failing in the field took

1000+ hours of instrumental exposure filtered by the thermal liner compared to more than fourteen weeks of natural sunlight. Therefore instrumental exposure should be used for its ability to produce the same results as the failed garments in the field at an accelerated exposure as compared to natural sunlight.

A test method should expose the moisture barrier as a single layer. Although the same color changes observed in failed garments from the field were seen in the threepiece ensembles, the severe cracking and flaking of the film was not apparent at these times of exposure. If the exposure would have continued beyond failing the Hydrostatic Water Penetration Resistance Test the cracking may have occurred. In comparison, the single moisture barriers did not show the same color changes but cracking and flaking of the films was apparent before leakage failure occurred with less exposure time. Also, the number of hours of exposure required before failure occurred in the three-piece ensemble samples was extensive. A test method that used the three-piece ensemble in the exposure, the time to reach failure would not only take several hundred hours but would also be expensive to conduct and replicate. Because of the length of time and the cost of testing using the three-piece ensemble, the researcher again recommends the test method specify exposing the single moisture barrier.

The results of this study produced inconclusive results for its investigation of abrasion by flexing as a pretreatment. The areas of extensive damage in the field garments were areas in which abrasion could be a contributing factor, especially in the pants used in the initial evaluation of garments from the field. Hence, the conclusion based on this study is that further investigation of the role of abrasion and light is needed.

The test method should include some type of pretreatment and/or post treatment that included textile-to-textile surface abrasion.

In the development of a test method, assessment parameters must include both visual and performance evaluations. The findings of this study strongly support the value of visual assessment throughout the treatments and exposures. Initially visual evaluations assessed the color as well as established the overall appearance of the film or coating's surface structure used in moisture barrier construction. Incremental visual assessments were somewhat predictive of performance. In some moisture barriers the appearance of cracks was in direct correlation to performance testing; that is cracks were always present when some types of moisture barriers failed the Hydrostatic Water Penetration Resistance Test.

Test method assessment should include a measure of performance and the Hydrostatic Water Penetration Resistance Test was an effective evaluation tool. The water penetration resistance test was a performance measurement used to assess leakage failure. This test is a NFPA requirement and the researcher concludes that it should be used as one measure of performance in the test method. The Hydrostatic Water Penetration Resistance Test or some other type of liquid penetration resistance testing should be used in the test method. This type of performance measurement will identify when failure has occurred and will allow for a prediction of failure in the field.

Limitations

One limitation of this study was the lack of replications in the natural sunlight exposure. Due to cost and time, only one replication per moisture barrier sample was evaluated for any given week. Another limitation of the natural sunlight treatment was

the ability to return samples to natural sunlight exposure if the sample passed the Hydrostatic Water Penetration Resistance Test. Also there were no failures seen in the three-piece ensembles due to the length of time of exposure.

Flexing in this study was conducted on all single moisture barriers, which did not allow for a comparison of un-flexed single moisture barriers. The only comparison between flexed and un-flexed samples were in the three-piece ensembles which were not enough to draw any conclusions as to whether flexing affected the degradation of the moisture barrier.

Another limitation to this study was the number of replications for the three-piece ensembles in the instrumental exposures. Only one replication of each three-piece ensemble was obtainable due to the time of exposure before failure. Although there was one preconditioned and one un-preconditioned three-piece ensemble that failed, there was no replication of a single sample. Therefore, the findings of this study are only representative of that sample.

This study was also limited to the number of turnout gear materials available for testing. The moisture barriers selected for this study may not represent all moisture barriers seen in today's fire fighting turnout gear.

Recommendations for Future Research

The recommendations for future research are based on the results of this study and could provide additional information for developing guidelines for a standard test method that could be used by NFPA Technical Committees for the assessment of moisture barriers.

Hydrostatic Water Penetration Resistance Test for 2 minutes at 2 psi: exposing the samples to the Hydrostatic Water Penetration Resistance Test for two minutes at 2 psi did the assessment for water permeability. To predict moisture barrier failure the NFPA 1971 standards specify the test to be measured for five minutes with 1 psi of pressure. It is recommended that future researchers use the NFPA conditions and evaluate the Hydrostatic Water Penetration Resistance Test for five minutes at 1 psi. NFPA 1971, 2000 Edition also requires the moisture barrier to resist viral and some liquid chemical challenges. A test method should include the resistance to viral and chemical liquids because they may be more challenging than water. For instance, during this study shallow cracks were apparent before the moisture barrier failed the water leakage test, but it is not known if it would have passed the viral penetration challenge.

Orientation of the Test Sample during Exposure to Light Source: The researcher recommends exposure of the single layer moisture barrier, however changing the orientation of the three-piece ensemble during exposure to the light source could be an area that merits further investigation. Samples in this study were exposed as three-piece ensembles and single layer moisture barriers. The three-piece ensembles were exposed with the thermal liner facing the light source of instrumental light exposure and the outer shell facing the light source during natural sunlight exposure. Obviously the moisture barrier is never exposed to direct light but in this study the film side of the moisture barrier was directly exposed. In the field, if light reaches the single moisture barrier it must pass through the outer shell or thermal liner. However, in this study the three-piece ensembles were exposed to light through the thermal liner in the instrumental exposure, but in both directions for natural sunlight exposure. This orientation of the sample was

also selected because garments from the field showed that failures of the moisture barrier occurred in areas where the thermal liner was predominately exposed to a light source during storage in the fire station, such as when the pant is rolled down around the boots.

Orientation of the Sample during Hydrostatic Water Penetration Resistance Testing: In this study, samples were evaluated with the film side of the moisture barrier facing down during the Hydrostatic Water Penetration Resistance Test. This required water to first pass through the film before penetrating the substrate. The current NFPA 1971 standards require the film side to be facing up on the water penetration resistance test, which allows water to flow through the substrate and simulating the garment as worn, simulating the orientation of the moisture barrier as it is worn in the field. It is recommended that future researchers use the NFPA requirements for the Hydrostatic Water Penetration Resistance NFPA 1971, 2000 Edition.

Extensive Analysis of Moisture Barrier Degradation: Further investigation of the degradation of the moisture barrier should be conducted. The researcher recommends, for example, that a chemical analysis of the polymers used in the film and the substrate be conducted, in addition to visual and water penetration assessments.

APPENDIX A

Table 1: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample A

Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial Evaluation	Pass	Gray	Small cell like, yellow and dark gray, some light pink areas, lighter in some areas, small craters
500*	Pass	Light blue in exposed area	Same as above
520	Pass	Same as above	Same as above
540	Pass	Light blue in exposed area	Same as above
560	Pass	Darker blue in exposed area	Same as above
580	Pass	Darker blue in exposed area	Same as above
600	Pass	Darker blue in exposed area	Same as above
620	Pass	Same as above	Light blue
640	Pass	Darker blue in exposed area	Blue/green with red splotches or spots
660	Pass	Same as above	Same as above
680	Pass	Darker blue	Same as above
700-740**	Pass	Same as above	Same as above
760	Pass	Darker blue	Same as above
780-800**	Pass	Same as above	Same as above
820	Pass	Same as above	Blue tint
840	Pass	Same as above	Same as above
860	Pass	Same as above	Bright blue with red splotches
880-1000**	Pass	Same as above	Same as above
1020	Pass	Darker blue	Darker blue tint
1040	Failed	Lighter blue	Same as above

* 0-480 was tested every 20-hours. Only a light change in color was seen. Results can be seen in Table 4.1

Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
			Small cell like, yellow and
Initial	Pass	Cross	dark gray, some light pink
Evaluation	Fass	Gray	areas, lighter in some areas,
			small craters
500*	Pass	Light gray in exposed area	Same as above
520	Pass	Same as above	Same as above
540	Pass	Very Light blue in exposed area	Lighter in exposed area
560	Pass	Lighter blue in exposed area	Same as above
580	Pass	Lighter in exposed area, light pink in hydrostatic test area	Same as above
600-620	Pass	Same as above	Same as above
640	Pass	Pink shading in exposed area	Dark spots
660-700**	Pass	Same as above	Same as above
720	Pass	Darker blue	Same as above
740	Pass	Same as above	Same as above
760	Pass	Darker blue with pink shading in exposed area	Same as above
780-800**	Pass	Same as above	Same as above
820	Pass	Same as above	Lighter gray/blue tint
840	Pass	Same as above	Light blue tint
860-880**	Pass	Same as above	Same as above
900	Pass	Blue/green with light pink shading	Light Yellow and blue/green tint with red splotches and dark spots
920	Pass	Light blue/green	Light green tint
940- 1000**	Pass	Same as above	Same as above
1020	Pass	Darker blue with pink shading in Hydrostatic Test area	Darker blue tint
1040	Pass	Darker blue color	Yellow/green tint with red splotches

Table 2: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample B

* 0-480 was tested every 20-hours. Only a light change in color was seen. Results can be seen in Table 4.2

Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1060	Pass	Same as above	Blue/green Tint
1080	Pass	Light Green	Green Yellow Tint
1100	Pass	Same as above	Same as above
1120	Pass	Darker Green with Gray shading	Green Tint
1140	Pass	Same as above	Light Green Tint
1160- 1200**	Pass	Same as above	Same as above
1220	Failed	Darker Green	Darker Green tint with dark spots

Table 2: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample B (continued)

* 0-480 was tested every 20-hours. Only a light change in color was seen. Results can be seen in Table 4.2

Hours of	Hydrostatic	Stereo Microscope	Compound
Exposure	Water Test	7X-35X	Microscope 100X
			Small cell like, yellow
Initial			and dark gray, some
Evaluation	Pass	Gray	light pink areas, lighter
L'unumon			in some areas, small
			craters
500*	Pass	Light blue in exposed area	Same as above
520	Pass	Can see Wrinkle Marks from Flexing	Same as above
540	Pass	Dark Blue in exposed area	Light Green tint with
			Red splotches
560-580**	Pass	Same as above	Same as above
600	Pass	Darker Blue in exposed area	Blue/green tint with
			Red splotches
620	Pass	Same as above	Blue tint with Red
640	D	Dedea Dia in a se i	splotches
640 660	Pass Pass	Darker Blue in exposed area	Same as above
000	Pass	Same as above	Same as above
680	Pass	Same as above	Dark Blue tint with
700	Pass	Same as above	Red splotches Same as above
			Darker Blue tint with
720	Pass	Same as above	Red splotches
740-860**	Pass	Same as above	Same as above
			Dark blue, Red
880	Pass	Same as above	splotches
900-920**	Pass	Same as above	Same as above
940	Pass	Same as above	Blue/green tint
960-980**	Pass	Same as above	Same as above
1000	Pass	Same as above	Dark Spots
1020-1220**	Failed	Same as above	Same as above

Table 3: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample C

* 0-480 was tested every 20-hours. Only a light change in color was seen. Results can be seen in Table 4.1

Table 4: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample D

ExposureWater Test7X-35X100XInitial EvaluationPassGraySmall cell like, ye dark gray, some li areas, lighter in so small crate500*PassLight blue in exposed area Same as aboveSame as above520PassSame as aboveSame as above540PassLight gray in exposed area Same as aboveSame as above560PassSame as aboveSame as above580PassLight ergray in exposed areaLighter gray in exposed area600-620**PassSame as aboveSame as above640PassLight blue in exposed area Same as aboveSame as above660PassSame as aboveSame as above680PassSame as aboveSame as above680PassSame as aboveSame as above700PassSame as aboveSame as above720PassSame as aboveSame as above740PassLight green with gray shading in exposed areaMore of a blue	roscope
Initial EvaluationPassGraydark gray, some li areas, lighter in sol small crate500*PassLight blue in exposed area Same as aboveSame as above520PassLight gray in exposed area Same as aboveSame as above540PassLight gray in exposed area areaSame as above560PassSame as aboveSame as above580PassLighter gray in exposed areaLighter gray and p600-620**PassSame as aboveSame as above640PassLight blue in exposed area areaSame as above640PassLight blue in exposed area Same as aboveSame as above640PassSame as aboveSame as above700 <td< th=""><th>•</th></td<>	•
EvaluationPassGrayareas, lighter in so small crate500*PassLight blue in exposed areaSame as above520PassSame as aboveSame as above540PassLight gray in exposed areaSame as above560PassSame as aboveSame as above580PassLighter gray in exposed areaLighter gray and p600-620**PassSame as aboveSame as above640PassLight blue in exposed areaSame as above660PassSame as aboveSame as above680PassSame as aboveSame as above680PassSame as aboveSame as above700PassSame as aboveSame as above720PassSame as aboveBlue/green/yellow red splotch740PassLight green with gray shading in exposed areaMore of a blue	
500*PassLight blue in exposed areasmall crate520PassSame as aboveSame as aboveSame as above540PassLight gray in exposed areaSame as aboveSame as above560PassSame as aboveSame as aboveSame as above580PassLighter gray in exposed areaLighter gray in exposed areaLighter gray and p600-620**PassSame as aboveSame as above640PassLight blue in exposed area Same as aboveSame as above660PassSame as aboveSame as above680PassSame as aboveSame as above680PassSame as aboveSame as above700PassSame as aboveSame as above720PassSame as aboveBlue/green/yellow red splotch740PassLight green with gray shading in exposed areaMore of a blue	• 1
500*PassLight blue in exposed areaSame as above520PassSame as aboveSame as above540PassLight gray in exposed areaSame as above560PassSame as aboveSame as above580PassLighter gray in exposed areaLighter gray and p600-620**PassSame as aboveSame as above640PassLight blue in exposed areaSame as above660PassSame as aboveSame as above660PassSame as aboveSame as above680PassSame as aboveSame as above680PassSame as aboveSame as above700PassSame as aboveSame as above720PassSame as aboveSame as above740PassLight green with gray shading in exposed areaMore of a blue	,
520PassSame as aboveSame as above540PassLight gray in exposed areaSame as above560PassSame as aboveSame as above580PassLighter gray in exposed areaLighter gray and p600-620**PassSame as aboveSame as above640PassLight blue in exposed areaSame as above640PassLight blue in exposed areaSame as above660PassSame as aboveSame as above680PassSame as aboveSame as above680PassSame as aboveSame as above700PassSame as aboveSame as above720PassSame as aboveBlue/green/yellow red splotch740PassLight green with gray shading in exposed areaMore of a blue	
540PassLight gray in exposed area Same as aboveSame as above Same as above560PassSame as aboveSame as above580PassLighter gray in exposed areaLighter gray and p600-620**PassSame as aboveSame as above640PassLight blue in exposed area Same as aboveSame as above660PassSame as aboveSame as above660PassSame as aboveSame as above680PassSame as aboveSame as above700PassSame as aboveSame as above720PassSame as aboveSame as above740PassLight green with gray shading in exposed areaMore of a blue	
560PassSame as aboveSame as above580PassLighter gray in exposed areaLighter gray and p600-620**PassSame as aboveSame as above640PassLight blue in exposed areaSame as above660PassSame as aboveSame as above660PassSame as aboveSame as above660PassSame as aboveSame as above660PassSame as aboveSame as above680PassSame as aboveSame as above680PassSame as aboveSame as above700PassSame as aboveSame as above720PassSame as aboveBlue/green/yellow red splotche740PassLight green with gray shading in exposed areaMore of a blue	
580PassLighter gray in exposed areaLighter gray and p600-620**PassSame as aboveSame as above640PassLight blue in exposed areaSame as above660PassSame as aboveSame as above680PassSame as aboveLight yellow gre with red splot700PassSame as aboveSame as above720PassSame as aboveBlue/green/yellow red splotche740PassLight green with gray shading in exposed areaMore of a blue/green/yellow red splotche	
380PassareaLighter gray and p600-620**PassSame as aboveSame as above640PassLight blue in exposed areaSame as above660PassSame as aboveSame as above680PassSame as aboveLight yellow gree680PassSame as aboveSame as above700PassSame as aboveSame as above720PassSame as aboveBlue/green/yellow red splotche740PassLight green with gray shading in exposed areaMore of a blue	ove
640PassLight blue in exposed areaSame as above660PassSame as aboveSame as above680PassSame as aboveLight yellow gree680PassSame as aboveSame as above700PassSame as aboveSame as above720PassSame as aboveBlue/green/yellow red splotche740PassLight green with gray shading in exposed areaMore of a blue	ink areas
660PassSame as aboveSame as above680PassSame as aboveLight yellow gre with red splot700PassSame as aboveSame as above720PassSame as aboveSame as above740PassLight green with gray shading in exposed areaMore of a blue	ove
680PassSame as aboveLight yellow grewith red splot700PassSame as aboveSame as above720PassSame as aboveBlue/green/yellow red splotch740PassLight green with gray shading in exposed areaMore of a blue	ove
680PassSame as abovewith red splot700PassSame as aboveSame as aboveSame as above720PassSame as aboveBlue/green/yellow red splotch740PassLight green with gray shading in exposed areaMore of a blue	ove
700PassSame as abovewith red splot720PassSame as aboveBlue/green/yellow red splotch740PassLight green with gray shading in exposed areaMore of a blue	en tint
720PassSame as aboveBlue/green/yellow red splotch740PassLight green with gray shading in exposed areaMore of a blue	ches
720PassSame as abovered splotch740PassLight green with gray shading in exposed areaMore of a blue	ove
740PassLight green with gray shading in exposed areaMore of a blue	
shading in exposed area	es
	e tint
	_
760PassSame as aboveBlue/Yellow tint d	*
780PassSame as aboveSame as above	
800 Pass Same as above Light blue/gree	
820PassSame as aboveSame as above	
840PassSame as aboveLight green	
860PassSame as aboveSame as above	
880 Pass Light blue/green Same as abo	
900PassSame as aboveSame as above	
920PassSame as aboveLight green – yel	
940PassSame as aboveSame as above	
960 Pass Same as above Lighter blue	
980 Pass Darker blue Same as abo	ove
1000- 1040**FailedSame as aboveSame as above	

* 0-480 was tested every 20-hours. Only a light change in color was seen. Results can be seen in Table 4.2

Table 5: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample B

Exposure	Hydrostatic	Stereo Microscope	Compound
Week	Water Test	7X-35X	Microscope 100X
			Small cell like, yellow
Initial			and dark gray, some
Evaluation	Pass	Gray	light pink areas, lighter
Lvaluation			in some areas, small
			craters
Week 1	Pass	Same as above	Dark red
Week 2	Pass	Grayish/Blue in color	Light red splotches
Week 3-4	Pass	Same as above	Same as above
Week 5	Pass	Bright blue in color	Blue tint
Week 6	Pass	Same as above	Dark blue tint
Week 7	Pass	Same as above	Greenish blue tint
Week 8	Pass	Same as above	Blue tint
Week 9	Pass	Same as above	Same as above
Week 10	Pass	Blue in color	Same as above
Week 11	Pass	Same as above	Dark and bright blue tint
Week 12	Pass	Same as above	Greenish/blue tint
Week 13	Pass	Same as above	Dark blue tint
Week 14	Pass	Same as above	Same as above

Table 6: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample N

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial Evaluation	Pass	Gray	Small cell like, yellow and dark gray, some light pink areas, lighter in some areas, small craters
Week 1	Pass	Same as above	Dark red
Week 2	Pass	Same as above	Yellow/gray tint
Week 3	Pass	Same as above	Bluish/yellow tint
Week 4	Pass	Same as above	Bluish/gray tint
Week 5	Pass	Light blue in color	Blue tint
Week 6 - 8	Pass	Same as above	Same as above
Week 9	Pass	Same as above	Dark blue tint
Week 10	Pass	Same as above	Same as above
Week 11	Pass	Blue with red splotches, some craters	Same as above
Week 12	Pass	Same as above	Same as above
Week 13	Pass	Bright blue with red splotches	Same as above
Week 14	Pass	Same as above	Same as above

Table 7: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample O

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial Evaluation	Pass	White, substrate is visible through film	Orange/yellow, lighter areas, craters
Week 1	Pass	Same as above	Dull yellow
Week 2	Pass	Light gray in color	Yellow with white areas
Week 3	Pass	Light brown	Same as above
Week 4 - 5	Pass	Same as above	Same as above
Week 6	Pass	Same as above	Yellow with Light lighter areas
Week 7 – 12	Pass	Same as above	Same as above
Week 13	Pass	Moderately brown in color	Same as above
Week 14	Pass	Same as above	Same as above

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial Evaluation	Pass	White, substrate is visible through film	Orange/yellow, lighter areas, craters
Week 1	Pass	Same as above	Dull yellow
Week 2	Pass	Light gray in color	Same as above
Week 3	Pass	Light brown	Yellow with bright white areas
Week 4 - 5	Pass	Same as above	Same as above
Week 6	Pass	Same as above	Yellow with Light lighter areas
Week 7	Pass	Same as above	Yellow with lighter areas
Week 8 - 11	Pass	Same as above	Same as above
Week 12	Pass	Darker brown in color	Same as above
Week 13 - 14	Pass	Same as above	Same as above

Table 8: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample P

Table 9: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample Q

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial Evaluation	Pass	White, wrinkled, substrate can be seen through film	Web-like pattern of fibers allowing light to pass through
Week 1 - 9	Pass	Same as above	Same as above
Week 10	Pass	Same as above	Bright yellow
Week 11 - 14	Pass	Same as above	Same as above

Table 10: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample R

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial		White, wrinkled,	Web-like pattern of
Evaluation	Pass	substrate can be seen	fibers allowing light
Evaluation		through film	to pass through
Week 1 - 9	Pass	Same as above	Same as above
Week 10	Pass	Same as above	Dark Yellow
Week 11 - 14	Pass	Same as above	Same as above

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial Evaluation	Pass	White, fibers seen under film	Web-like pattern of fibers, light areas
Week 1 - 9	Pass	Same as above	Same as above
Week 10	Pass	Light Brown	Dark Yellow
Week 11 - 14	Pass	Same as above	Same as above

Table 11: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample S

Table 12: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample T

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X	
Initial Evaluation	Pass	White, fibers seen under film	Web-like pattern of fibers, light areas	
Week 1 - 7	Pass	Same as above	Same as above	
Week 8	Pass	Light Pink with craters	Same as above	
Week 9	Pass	Same as above	Same as above	
Week 10	Pass	Light brown	Same as above	
Week 11 - 14	Pass	Same as above	Same as above	

Table 13: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample U

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial Evaluation	Pass	Yellow/white very wrinkled film	Grainy, bright yellow, lighter areas
Week 1 - 4	Pass	Same as above	Same as above
Week 5	Pass	Light brown	Same as above
Week 6 – 8	Pass	Same as above	Same as above
Week 9	Pass	Light yellow	Same as above
Week 10 - 13	Pass	Same as above	Same as above
Week 14	Pass	Moderately Brown	Same as above

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X	
Initial Evaluation	Pass	Yellow/white very wrinkled film	Grainy, bright yellow, lighter areas	
Week 1	Pass	Same as above	Same as above	
Week 2	Pass	Light brown	Same as above	
Week 3 – 6	Pass	Same as above	Same as above	
Week 7	Pass	Darker brown	Same as above	
Week 8 - 9	Pass	Same as above	Same as above	
Week 10	Pass	Light yellow	Same as above	
Week 11	Pass	Moderately Brown	Same as above	
Week 12 - 14	Pass	Same as above	Same as above	

Table 14: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample V

Table 15: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample W

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X	
Initial	Pass	White, substrate is	Orange/yellow, lighter	
Evaluation	Pass	visible through film	areas, craters	
Week 1	Pass	Same as above	Same as above	
Week 2	Pass	Light gray	Same as above	
Week 3 – 5	Pass	Same as above	Same as above	
Week 6	Pass	Light brown	Same as above	
Week 7 - 8	Pass	Same as above	Same as above	
Week 9	Pass	Same as above	Yellow with lighter areas	
Week 10	Pass	Same as above	Same as above	
Week 11	Pass	Darker brown	Same as above	
Week 12	Pass	Same as above	Same as above	
Week 13	Pass	Moderately Brown	Same as above	
Week 12 - 14	Pass	Same as above	Same as above	

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial Evaluation	Pass	White, fibers seen under film	Web-like pattern of fibers, light areas
Week 1	Pass	Same as above	Same as above
Week 2	Pass	Light gray	Same as above
Week 3	Pass	Light brown	Same as above
Week 4 - 12	Pass	Same as above	Same as above
Week 13	Pass	Moderately brown	Same as above
Week 14	Pass	Same as above	Same as above

Table 16: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample X

APPENDIX B

Table 17: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample E

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1	Initial Evaluation	Pass	White, substrate is visible through film	Orange/yellow, lighter areas, craters
1	20	Pass	Whiter with light yellow shading in exposed area	Same as above
1	40	Pass	Whiter in exposed area	Same as above
1	60-140*	Pass	Same as above	Same as above
1	160	Pass	Whiter in Hydrostatic Test Area	Begin to see fibers under film, becoming web- like
1	180	Pass	Same as above	White and dark yellow areas
1	200	Failed	Same as above	Same as above
2	Initial Evaluation	Pass	White, substrate is visible through film	Orange/yellow, lighter areas, craters
2	20	Pass	Whiter in exposed area	Dark yellow, begin to see fibers
2	40	Pass	Same as above	Dark yellow with white areas
2	60-100*	Pass	Same as above	Same as above
2	120	Pass	Same as above	Fibers are becoming bright
2	140	Pass	Starting to yellow	Same as above
2	160	Pass	Light yellow	Same as above
2	180	Failed	Same as above	Same as above
3	Initial Evaluation	Pass	White, substrate is visible through film	Orange/yellow, lighter areas, craters
3	20	Pass	Whiter in exposed area	Dark yellow can barely see fibers
3	40-60*	Pass	Same as above	White areas
3	80	Pass	Same as above	More white areas
3	100-120*	Pass	Same as above	Same as above

Table 17: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample E (continued)

Replication	Hours of	Hydrostatic	Stereo Microscope	Compound
Replication	Exposure	Water Test	7X-35X	Microscope 100X
3	140	Pass	Same as above	More white areas
			Same as above	than yellow areas
3	160-200*	Failed	Same as above	Same as above
	Initial	D.	White, substrate is	Orange/yellow,
4	Evaluation	Pass	visible through film	lighter areas,
				craters
4	20	Pass	Whiter in exposed	Dark yellow with white areas, begin
	20	1 455	area	to see fibers
4	40-80*	Pass	Same as above	Same as above
4	100	Pass	Same as above	More white areas
				Lighter yellow,
4	120	Pass	Same as above	begin to see more
				fibers
4	140-160*	Pass	Same as above	Same as above
				Areas that look
4	180	Failed	Same as above	like forming
				cracks

Table 18: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc	
Exposure - Sample F	

Replication	Hour of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1	Initial Evaluation	Pass	White, wrinkled, substrate can be seen through film	Web-like pattern of fibers allowing light to pass through
1	20	Pass	Tan color in exposed area	Same as above
1	40-60*	Pass	Same as above	Same as above
1	80	Pass	Tan color in exposed area	Same as above
1	100	Pass	Same as above	Same as above
1	120	Pass	Brown in exposed area	Same as above
1	140 160	Pass Failed	Same as above Darker brown in exposed area, Lighter in Hydrostatic Test area	Same as above Same as above
2	Initial Evaluation	Pass	White, wrinkled, substrate can be seen through film	Web-like pattern of fibers allowing light to pass through
2	20	Pass	Tan color in exposed area	Same as above
2	40	Pass	Darker tan color	Same as above
2	60	Pass	Same as above	Same as above
2	80	Pass	Darker tan color in exposed area	Can see more fibers
2	100	Pass	Same as above Dark tan, lighter in	Same as above
2	120	Pass	Hydrostatic Test area	Same as above
2	140	Pass	Film is wearing	Same as above
2	160-180*	Failed	Same as above	Dark yellow areas

*Samples were evaluated every 20-hours. No change was seen at each 20-hour increment.

Replication	Hour of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
3	Initial Evaluation	Pass	White, wrinkled, substrate can be seen through film	Web-like pattern of fibers allowing light to pass through
3				
3	20	Pass	Moderate tan	Dark yellow, dark spots
3	40	Pass	Darker tan in color	Darker yellow
3	60	Pass	Darker tan in color	White areas
3	80	Pass	Same as above	Dense web pattern of fibers, larger white areas, tan/yellow in color
3	100	Pass	Darker tan in exposed area Lighter in	Same as above
3	120	Pass	hydrostatic test area	Same as above
3	140-160*	Pass	Same as above	Same as above
3	180	Pass	Brown in exposed area, lighter in Hydrostatic Test area	Same as above
3	200	Failed	Film is wearing	Same as above
4	Initial Evaluation	Pass	White, wrinkled, substrate can be seen through film	Web-like pattern of fibers allowing light to pass through
4	20	Pass	Tan in exposed area	Dark yellow, dark and light areas
4	40	Pass	Same as above	White areas
4	60	Pass	Same as above hange was seen at eac	Same as above

Table 18: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample F (continued)

*Samples were evaluated every 20-hours. No change was seen at each 20-hour increment

Replication	Hour of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
4	80	Pass	Dark tan in exposed area, lighter in Hydrostatic Test area	Same as above
4	100	Pass	Same as above	Same as above
4	120	Pass	Brown in exposed area, lighter in Hydrostatic Test area	Same as above
4	140	Pass	Same as above	Thin areas appear in the film
4	160	Pass	Same as above	Dense web- like pattern of fibers, bright fibers
4	180-200*	Failed	Same as above	Same as above

Table 18: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample F (continued)

*Samples were evaluated every 20-hours. No change was seen at each 20-hour increment

Table 19: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample G

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1	Initial Evaluation	Pass	Gray	Small cell like, yellow and dark gray, some light pink areas, lighter in some areas, some small craters
1	20	Pass	Very Light blue in exposed area	Greenish-yellow in exposed area
1	40	Pass	Very Light blue in exposed area	Same as above
1	60	Pass	Lighter blue in exposed area	Same as above
1	80	Pass	White in exposed area	Same as above
1	100	Failed	Same as above	Small cracks
2	Initial Evaluation	Pass	Gray	Small cell like, yellow and dark gray, some light pink areas, lighter in some areas, some small craters
2	20	Pass	Very Light blue in exposed area	Same as above
2	40	Pass	Light blue in exposed area	Blue/green tint, red splotches
2	60	Pass	Same as above Light blue to	Same as above
2	80	Pass	white in exposed area	Can see cracks
2	100	Failed	Same as above	Can see holes and cracks

Table 19: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample G (continued)

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X- 35X	Compound Microscope 100X
3	Initial Evaluation	Pass	Gray	Small cell like, yellow and dark gray, some light pink areas, lighter in some areas, some small craters
3	20	Pass	Very Light blue in exposed area	Same as above
3	40	Pass	Light gray in exposed area	Blue/green tint, red splotches
3	60	Pass	Same as above Light blue to	Same as above
3	80	Pass	white in exposed area, can see cracks	Same as above
3	100	Failed	Light White in exposed area	Can see small cracks, lighter, Blue/green tint
4	Initial Evaluation	Pass	Gray	Small cell like, yellow and dark gray, some light pink areas, lighter in some areas, some small craters
4	20	Pass	Light gray in exposed area	Same as above
4	40	Pass	Same as above	Greenish-yellow tint, red splotches
4	60	Pass	Light gray in exposed area	Same as above
4	80	Pass	Light gray to with light yellow in exposed area	Same as above
4	100	Failed	Same as above	Can see small cracks, Lighter Greenish- yellow tint

Table 20: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample H

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1	Initial Evaluation	Pass	Yellow/white, areas that look like forming craters	Yellow/light orange, grainy, craters, lighter areas
1	20	Pass	Loss of yellow in exposed area	Yellow-orange in exposed area
1	40	Pass	Light pink in exposed area	Yellow in exposed area
1	60	Pass	Tan color in exposed area	Same as above
1	80	Pass	Same as above	Same as above
1	100	Pass	Darker tan color in exposed area	Same as above
1	120	Pass	Tan in exposed area	Same as above
1	140	Pass	White in exposed area	Same as above
1	160	Failed	Same as above	Cracks in failure area (observed after Hydrostatic Testing)
2	Initial Evaluation	Pass	Yellow/white, areas that look like forming craters	Yellow/light orange, grainy, craters, lighter areas
2	20	Pass	Loss of yellow (white) in exposed area	Yellow-orange in exposed area
2	40	Pass	Light tan in exposed area	Yellow in exposed area
2	60	Pass	Becoming brittle like**	Same as above
2	80	Pass	Same as above	Same as above
2	100	Pass	Darker tan color in exposed area	Same as above
2	120-140	Pass	Same as above	Same as above
2	160	Pass	Same as above	Dark yellow

*Samples were evaluated every 20-hours. No change was seen at each 20-hour increment.

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
2	180	Pass	Same as above	Can see cracking
2	200	Failed	Same as above	More cracking
3	Initial Evaluation	Pass	Yellow/white, areas that look like forming craters	Yellow/light orange, grainy, craters, lighter areas
3	20	Pass	Very light tan in exposed area	Dark yellow
3	40	Pass	Light tan in exposed area	Darker yellow
3	60-80	Pass	Same as above Lighter in	Same as above
3	100	Pass	Hydrostatic Test area	Same as above
3	120	Pass	Whiter in exposed area	Same as above
3	140	Pass	Same as above	Cracks are forming
3	160	Pass	Light gray shading	Same as above
3	180	Pass	Same as above	More cracking
3	200	Pass	Same as above	Same as above
3	220	Pass	Whiter in exposed area	Dark yellow
3	240	Failed	Same as above	More cracking
4	Initial Evaluation	Pass	Yellow/white, areas that look like forming craters	Yellow/light orange, grainy, craters, lighter areas
4	20	Pass	Very light tan in exposed area	Dark yellow
4	40	Pass	Same as above	Darker yellow
4	60	Pass	White in exposed area	Cracks are forming
4	80-100*	Pass	Same as above	Same as above
4	120	Pass	Same as above	Larger cracks
4	140-160*	Pass	Same as above	Same as above
4 *Samples were	180	Pass	Same as above	More cracking

*Samples were evaluated every 20-hours. No change was seen at each 20-hour increment.

Table 21: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample H (continued)

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
4	200-220*	Pass	Same as above	Same as above
4	240	Failed	Same as above	More cracking

*Samples were evaluated every 20-hours. No change was seen at each 20-hour increment.

Table 21: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample I

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1	Initial Evaluation	Pass	Cream color, very shiny	Red, some craters large and small, some lighter areas
1	20	Pass	Medium brown in exposed area, becoming brittle*, small craters, shiny	Same as above
1	40	Pass	Lighter brown in Hydrostatic Test area, cracks in exposed area, scorched smell	Areas that resemble sun spots, cracks
1	60	Pass	Getting darker in exposed area, can see wrinkles marks from flexing	Same as above
1	80	Pass	Lighter in Hydrostatic Test area, darker in other exposed areas	Same as above
1	100	Pass	More wrinkle marks from flexing	Same as above
1	120	Pass	Darker brown in exposed area	Same as above
1	140	Failed	Around Hydrostatic Test area the lighter color is spreading	Same as above
2	Initial Evaluation	Pass	Cream color, very shiny	Red, some craters large and small, some lighter areas
2	20	Pass	Medium Brown in exposed area, Becoming Brittle*	Same as above
2	40	Pass	Lighter in Hydrostatic Test area, scorched smell	Cracking

Table 21: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample I (continued)

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
2	60	Pass	Cracking, can see wrinkles from flexing (color change)	Same as above
2	80	Pass	Lighter color in Hydrostatic Test area is spreading to other areas, more cracking	More cracking
2	100	Pass	Dark brown color in exposed area, more cracking	More cracking
2	120	Pass	More cracking	More cracking
2	140	Pass	Same as above	Same as above
2	160	Pass	Darker brown in exposed area	Same as above
2	180	Failed	Dark brown in color, light tan in color in Hydrostatic Test area, can see black spots and more cracking	Deeper cracking, can see black spots
Initial Evaluation	Pass	Cream color, very shiny	Red, some craters large and small, some lighter areas	
3	20	Pass	Medium brown in exposed area, becoming brittle*, light scorched smell	Black spots
3	40	Pass	Lighter in Hydrostatic Test area	Cracking and holes, orange, white and black spots
3	60	Pass	Darker brown	More cracking and spots
3	80	Pass	Discoloration in wrinkled areas from flexing	More cracking

Table 21: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample I (continued)

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
3	100	Failed	Very Brittle*, very dark brown lighter in Hydrostatic Test Area	Same as above
Initial Evaluation	Pass	Cream color, very shiny	Red, some craters large and small, some lighter areas Medium brown in	
4	20	Pass	exposed area, becoming brittle*	Same as above
4	40	Pass	Dark brown in exposed area, cracking	Cracking
4	60	Pass	Lighter color in Hydrostatic Test area, scorched smell, can see wrinkles from flexing (color change)	Same as above
4	80	Pass	More cracking	More cracking
4	100	Pass	More cracking	More Cracking
4	120	Pass	More cracking	More Cracking
4	140	Failed	Same as above	Same as above

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1	Initial Evaluation	Pass	Yellow, raised and lowered surface on film	Grainy, bright yellow, lighter areas, web like pattern of fibers
1	20	Failed	Light brown in exposed area	Same as above
2	Initial Evaluation	Pass	Yellow, raised and lowered surface on film	Grainy, bright yellow, lighter areas, web like pattern of fibers
2	20	Pass	Light brown in exposed area	Same as above
2	40	Failed	Dark yellow color in exposed area	Same as above
3	Initial Evaluation	Pass	Yellow, raised and lowered surface on film	Grainy, bright yellow, lighter areas, web like pattern of fibers
3	20	Pass	Dark yellow color in exposed area	Same as above
3	40	Failed	Same as above	Same as above
4	Initial Evaluation	Pass	Yellow, raised and lowered surface on film	Grainy, bright yellow, lighter areas, web like pattern of fibers
4	20	Pass	Same as above	Same as above
4	40	Failed	Dark yellow color in exposed area	Same as above

Table 22: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample J

Table 23: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample K

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1	Initial Evaluation	Pass	Yellow, raised and lowered surface on film, can see fiber under film	Yellow, lighter area
1	20	Failed	Tan color in exposed area	Same as above
2	Initial Evaluation	Pass	Yellow, raised and lowered surface on film, can see fiber under film	Yellow, lighter area
2	20	Failed	Dark yellow color in exposed area	Same as above
3	Initial Evaluation	Pass	Yellow, raised and lowered surface on film, can see fiber under film	Yellow, lighter area
3	20	Failed	Dark yellow color in exposed area	Same as above
4	Initial Evaluation	Pass	Yellow, raised and lowered surface on film, can see fiber under film	Yellow, lighter area
4	20	Failed	Dark yellow color in exposed area	Same as above

Table 24: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample L

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1	Initial Evaluation	Pass	White, fibers seen under film	Web-like pattern of fibers, light areas
1	20	Pass	Pink in exposed area	Dark yellow in exposed area
1	40	Pass	Darker pink in exposed area	Film worn in small area from flexing, small crater
1	60	Pass	Darker pink in exposed area	Yellow with red shading in exposed area
1	80	Pass	Same as above	Same as above
1	100	Failed	Brittle* in exposed area	Same as above
2	Initial Evaluation	Pass	White, Fibers seen under film	Web-like pattern of fibers, light areas
2	20	Pass	Pink in exposed area	Dark yellow in exposed area
2	40	Pass	Darker pink in exposed area	Same as above
2	60	Failed	Darker pink in exposed area	Lighter and dark yellow shading
3	Initial Evaluation	Pass	White, fibers seen under film	Web-like pattern of fibers, light areas
3	20	Pass	Pink in exposed area	Dark yellow in exposed area
3	40	Pass	Darker pink in exposed area, becoming brittle*	Same as above
3	60	Pass	Darker pink in exposed area	Light and dark yellow shading
3	80	Failed	Light white in Hydrostatic Test area	Same as above

Table 24: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample L (continued)

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X- 35X	Compound Microscope 100X
4	Initial Evaluation	Pass	White, fibers seen under film	Web-like pattern of fibers, light areas
4	20	Pass	Pink in exposed area	Same as above
4	40	Pass	Same as above	Dark yellow tint
4	60	Pass	Same as above	Same as above
4	80	Pass	Light white in Hydrostatic Test area	Same as above
4	100	Failed	Same as above	Same as above

Table 25: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample M

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
1	Initial Evaluation	Pass	Yellow/white very wrinkled film	Grainy, bright yellow, lighter areas
1	20	Pass	Tan color in exposed area	Same as above
1	40	Pass	Light pink in exposed area	Craters
1	60	Pass	Same as above	Same as above
1	80	Pass	Tan in exposed area	Same as above
1	100	Pass	Same as above	Same as above
1	120	Pass	Starting to yellow	Same as above
1	140	Pass	Same as above	Same as above
	160	Pass	Yellow/tan in color in exposed area	Same as above
1	180	Failed	Same as above	Same as above
2	Initial Evaluation	Pass	Yellow/white very wrinkled film	Grainy, bright yellow, lighter areas
2	20	Pass	Light yellow in exposed area	Same as above
2	40	Pass	Light tan in exposed area	Same as above
2	60	Pass	Tan in exposed area	Same as above
2	80	Pass	Darker tan in exposed area	Dark yellow, forming craters
2	100	Pass	Tan/yellow in exposed area	Same as above
2	120	Pass	Dark yellow in exposed area	Same as above
2	140-160*	Failed	Same as above	Same as above
3	Initial Evaluation	Pass	Yellow/white very wrinkled film	Grainy, bright yellow, lighter areas
3	20	Pass	Light tan in exposed area	Lighter yellow

*Samples were evaluated every 20-hours. No change was seen at each 20-hour increment.

Table 25: Microscopic Evaluations and Hydrostatic Water Test Results for Carbon Arc Exposure - Sample M (continued)

Replication	Hours of Exposure	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
3	40	Pass	Darker tan in exposed area	Dark yellow
3	60-80*	Pass	Same as above	Same as above
33	100	Pass	Yellow in exposed area	Same as above
3	120	Pass	Same as above	Same as above
3	140	Failed	Light yellow in exposed area	Same as above
4	Initial Evaluation	Pass	Yellow/white very wrinkled film	Grainy, bright yellow, lighter areas
4	20	Pass	Moderate yellow in exposed area	Brighter yellow
4	40	Pass	Light tan in exposed area	Same as above
4	60	Pass	Yellow/tan in exposed area	Same as above
4	80	Pass	Same as above	Light yellow
4	100	Pass	More yellow in exposed area	Same as above
4	120	Pass	Same as above	Same as above
4	140	Failed	Light yellow	Same as above

*Samples were evaluated every 20-hours. No change was seen at each 20-hour increment.

Exposure	Hydrostatic	Stereo Microscope	Compound Microscope
Week	Water Test	7X-35X	100X
Initial	Pass	White, substrate is visible	Orange/yellow, lighter
Evaluation		through film	areas, craters
Week 1	Pass	Same as above	Same as above
Week 2	Pass	Light yellow	Bright yellow with light
WEEK 2	r ass	Light yellow	orange areas
Week 3	Pass	Light brown	Same as above
Week 4	Pass	Same as above	Same as above
Week 5	Pass	Same as above	Dark orange
Week 6 - 7	Pass	Same as above	Same as above
Week 8	Pass	Same as above	Dark orange with red and
WEEK O	F 455	Same as above	yellow areas
Week 9 - 13	Pass	Same as above	Same as above
Week 14	Pass	Darker brown	Same as above

Table 26: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample E

Exposure	Hydrostatic	Stereo Microscope	Compound Microscope
Week	Water Test	7X-35X	100X
Initial	Pass	White, wrinkled,	Web-like pattern of fibers
Evaluation	1 455	substrate can be seen	allowing light to pass
Evaluation		through film	through
Week 1	Pass	Same as above	Same as above
Week 2	Pass	Light yellow, fibers from substrate are dark orange	Same as above
Week 3	Pass	White with brown fibers	Same as above
Week 4	Pass	Same as above	Dark yellow with lighter areas
Week 5	Failed	Same as above	Same as above
Week 6	Pass	Light brown	Dark yellow
Week 7	Pass	Same as above	Yellow/orange with yellow areas
Week 8 – 9	Pass	Same as above	Same as above
Week 10	Pass	Delaminating film from substrate	Same as above
Week 11	Pass	Moderately brown	Same as above
Week 12	Pass	Same as above	Same as above
Week 13	Pass	Delaminating film from substrate	Same as above
Week 14	Pass	Same as above	Same as above

Table 27: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample F

Exposure	Hydrostatic	Stereo Microscope	Compound Microscope
Week	Water Test	7X-35X	100X
Initial Evaluation	Pass	Gray	Small cell like, yellow and dark gray, some light pink areas, lighter in some areas, some small craters
Week 1	Pass	Yellow in color	Yellow, cracks
Week 2	Pass	Darker yellow	Yellow/green, more cracking
Week 3	Failed	Cracking	Darker cells, more cracking
Week 4	Failed	More cracking, becoming brittle*	More cracking
Week 5	Failed	Same as above	Same as above
Week 6	Failed	More cracking	More cracking
Week 7 - 10	Failed	Same as above	Same as above
Week 11	Failed	More cracking, flaking of film	Same as above
Week 12	Failed	Same as above	Same as above
Week 13	Failed	More cracking and flaking of film	More cracking
Week 14	Failed	Same as above	Same as above

Table 28: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample G

Exposure	Hydrostatic	Stereo Microscope	Compound Microscope
Week	Water Test	7X-35X	100X
Initial	Pass	White, fibers seen	Web-like pattern of fibers,
Evaluation		under film	light areas
Week 1	Pass	Light yellow in color	Same as above
Week 2	Pass	Same as above	Dark orange
Week 3	Pass	Light brown	Dark yellow with orange areas
Week 4	Pass	Light pink	Same as above
Week 5 - 8	Pass	Same as above	Same as above
Week 9	Pass	Light brown	Same as above
Week 10	Pass	Delaminating of film from substrate	Same as above
Week 11	Pass	Moderately brown	Same as above
Week 12 - 13	Failed	Same as above	Same as above
Week 14	Pass	Same as above	Same as above

Table 29: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample L

Exposure	Hydrostatic	Stereo Microscope	Compound Microscope
Week	Water Test	7X-35X	100X
Initial	Pass	Yellow/white very	Grainy, bright yellow, lighter
Evaluation		wrinkled film	areas
Week 1	Pass	Dull yellow in color	Same as above
Week 2	Pass	Same as above	Same as above
Week 3	Pass	Bright yellow	Same as above
Week 4	Pass	Brittle*	Cracking and craters
Week 5	Pass	Cracking	Dark grains, cracking
Week 6	Failed	More cracking	More cracking
Week 7	Failed	Same as above	Same as above
Week 8	Pass	Same as above	Same as above
Week 9	Failed	Same as above	Same as above
Week 10	Failed	Yellow with white areas, more cracking	More cracking
Week 11	Failed	More cracking	Orange, more cracking
Week 12	Failed	Yellow with darker yellow areas, more cracking	More cracking
Week 13	Failed	Lighter and darker yellow, more cracking	More cracking
Week 14	Failed	Same as above	Same as above

Table 30: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample M

Exposure Week	Hydrostatic Water Test	Stereo Microscope 7X-35X	Compound Microscope 100X
Initial Evaluation	Pass	White	Small cell like, lighter in some areas, some small craters
Week 1	Pass	Yellow in color	Dark cells, dark yellow
Week 2	Failed	Same as above	Cracking
Week 3	Failed	Cracking	More cracking
Week 4	Failed	More cracking	More cracking
Week 5	Failed	Dark orange spots and cracking	More cracking
Week 6	Failed	More cracking, becoming brittle*	More cracking
Week 7	Failed	Bright yellow with brown spots, more cracking	Same as above
Week 8	Failed	No brown spots, more cracking	More cracking
Week 9	Failed	Same as above	Same as above
Week 10	Failed	More cracking and flaking of film	More cracking
Week 11	Failed	Same as above	Same as above
Week 12	Failed	Same as above	More cracking
Week 13	Failed	More cracking and flaking of film	More cracking
Week 14	Failed	More cracking and flaking of film	More cracking

Table 31: Microscopic Evaluations and Hydrostatic Water Test Results for Natural Sunlight Exposure - Sample Y

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