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Effect of humidity on accelerated aging of medical device pouches

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EFFECT OF HUMIDITY ON ACCELERATED AGING OF MEDICAL DEVICE POUCHES

A Thesis
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The Faculty of the Departments of Nutrition and Food Science
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In Partial Fulfillment
Of the Requirements for the Degree
Master of Science

By
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ABSTRACT

EFFECT OF HUMIDITY ON ACCELERATED AGING OF MEDICAL DEVICE POUCHES

By James Robert Wise

Tyvek®/polyester-polyethylene laminate pouches with water base adhesive were subjected to an accelerated aging test at the temperature levels of 45°C, 55°C, and 65°C (ambient humidity) for 1 and 2 year shelf lives. In addition, the accelerated aging tests were conducted at these three temperature levels simultaneously at elevated humidity conditions of 75% RH. The purpose of this research was to identify the effect of humidity in the accelerated aging process of medical device pouch package systems.

The data resulted in no significant difference from the high humidity test input. The test results showed that time was the leading factor in the degradation of the medical device pouch heat seals evaluated. In addition, the findings showed that the current industry practice of only recording peak force may be an over estimate of the seal strength. The mean propagation force was noted to give a truer representation of the heat seal characteristics.

ACKNOWLEDGEMENTS

I would like to express my deep appreciation to my graduate advisor, Dr. Jorge Marcondes. Jorge's dedication and perseverance has guided me through the process of obtaining a Masters of Science Degree. He is a very positive individual who conveyed interest and encouragement through the lengthy process to complete this work. Without his influence it would not be possible. For all of its value, this paper is dedicated to my good friend Jorge.

Herb Schueneman contributed greatly to the completion of this paper. Herb never blinked an eye to donate test equipment and resources at Westpak, Inc. and offered to be my SJSU liaison after I moved. Thanks are also extended to Dr. Lucy McProud for her technical review and support, [especially her willingness to increase her responsibility to the committee chair]. She is well respected and appreciated by many students who have ventured through the process of completing a master's thesis.

Many thanks are extended to Dr. Albert Elboudwarej for contributing to the technical content and validity of the work. Dr. Aharon Hibshoosh, thank you for participating and being the link between the packaging program and the business department. The material for this research was donated by Mr. Grey Tilley, Tolas Health Care Packaging. Thank you Grey for many lengthy discussions and support along the way.

The completion of this research would not have meaning without the loving support and help from my wife and daughter.

PREFACE

The following is a publication style thesis. The third chapter is written in journal format and will be submitted to the *Pharmaceutical & Medical Packaging News* Journal. Chapters 1, 2, and 4 are written according to the guidelines outlined in the *Publication Manual of the American Psychological Association*, 5th edition, 2001.

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List of Acronyms and Abbreviations

Accelerated aging (AA)	storage of samples at an elevated temperature (TAA) in order to simulate real time aging in a reduced amount of time
Accelerated aging factor (AAF)	an estimated or calculated ratio of the time to achieve the same level of physical property change as a package stored at real time (RT) conditions
Accelerated aging temperature (T_{AA})	the elevated temperature used to conduct the aging studies, and it is based on the ambient temperature or the estimated temperature of usage, or storage of this package, or both.
Accelerated aging time (AAT)	the length of time at which the accelerated aging is conducted
Ambient temperature (T_{RT})	storage temperature for real-time aging (RT) samples that represents storage conditions.
Crosshead Rate	separation rate of the universal test machine used to peel the specimens
Package shelf life	the amount of real time that a package can be expected to remain in storage at ambient conditions, or under specified conditions of storage, and maintain its critical performance properties
Real-time aging (RT)	storage time of samples at ambient conditions
Real-time equivalent (RTE)	amount of real-time aging to which given accelerated aging conditions are estimated to be equivalent
Zero time (t_0)	the beginning of an aging study

List of Variables

AAF	$Q_{10}\ [T_{AA} - T_{RT}/10]$
Accelerated Aging Time	RT (days)/ AAF
AAF	Accelerated Aging Factor
T_{AA}	Accelerated Aging Temperature
Q_{10}	2.0 (conservative coefficient for polymeric systems)
T_{RT}	Ambient Real-Time Storage Temperature
RT	Real-Time Shelf Life
msec	one thousandth of a second
lbf	Force pounds
Q_{10}	an aging factor for 10°C increase or decrease in
ipm	Inches per minute

CHAPTER 1

INTRODUCTION AND REVIEW OF LITERATURE

Introduction

Packaging is an essential component of the vast and complex distribution system. The package/product system should be designed to withstand the rigors of the distribution environment. From the point of the product's creation, the package system must survive the hazards encountered until it reaches its final destination and is finally utilized (Brandenburg & Lee, 1993).

Pharmaceutical and medical device industries are regulated by the Food and Drug Administration (FDA) and face strict requirements in order to validate their package systems and ship their product to the market. The medical device industry faces many packaging challenges, because their products are intended for operating room environments and are used as surgical tools or implants. The package system must be able to survive the stress of the sterilization process, hazards of the distribution cycle, the effects of time and temperature in storage, and ultimately introduce a sterile medical device to a surgical team. Any compromise to the sterile integrity of the package system is not acceptable and places human life and public safety at immediate risk.

The FDA requires that a shelf life must be declared for a medical device package system. This requirement is accomplished by conducting real-time shelf life testing. In order to ensure public safety and well being a process was created to speed the innovation of medical technology to the market.

Accelerated aging studies subject the package to elevated temperature and time inputs in order to simulate real-life aging. Industry consensus test standards guide and recommend procedures and methods for accelerated aging studies that meet the medical device requirements.

In 1998 the FDA recognized the official guidance standard ISO 11607: 1997 "Packaging for Terminally Sterilized Medical Devices" and in 1999 the American Society for Testing and Materials (ASTM) published ASTM F 1980-99 "Guide for Accelerated Aging of Sterile Medical Device Packages." These consensus standard guides provide the necessary framework for individual device manufacturers to develop and justify accelerated aging protocols to the FDA. The ASTM guide is the most current and well-developed reference that enables companies to write their own individual accelerated aging protocols. (Swain, 2000)

Medical device manufacturers commonly model their accelerated aging test protocol based on the Q_{10} theory of increased molecular activity reactions at elevated temperatures. With a push to get to the market faster, manufacturers are increasing the accelerated aging temperature of the Q_{10} formula in order to decrease the accelerated aging test duration. Test temperatures and durations are inversely related; a temperature increase of 10°C reduces the test duration by a factor of 2. Temperatures of 60°C, 65°C, 70°C and 75°C are being used in an attempt to speed up the process. Temperature inputs greater than 55°C are questionable and possibly unreliable. (Nolan, 2000)

Research Focus

The focus of this research is to determine the degradation effects that humidity has on the peel strength characteristics of a four-side sealed Tyvek®/polyester-polyethylene laminate pouch. This research is focused on the package system and not the interior contents. This is vital because companies are using "in package" shelf life tests to validate products. The effects of the temperature and humidity on the package system need to be accounted for.

The seal strength is not expected to have greater variations for tests with a humidity input as opposed to the same test without humidity. The humidity is not likely to be an added stress factor that will directly affect the seal strength properties. However, the properties of the materials are likely to become brittle and less elastic and increase the force necessary to separate the bond of the peel-able seal over the course of the accelerated aging process.

This research provides a benchmark for future medical device package systems. This benchmark will enable medical device manufacturers to understand the affect, if any, that humidity plays in the package shelf-life study process. This study may help companies reduce the quantity and cost of validation services if high humidity test inputs can be removed from the validation protocol.

Review of Literature

Accelerated Aging

Medical device manufacturers now have a standard to follow when conducting accelerated aging tests for their package systems. ASTM F1980-99 was ASTM's answer to creating a consensus test standard, which addressed accelerated aging for the medical device industry. The historical origin of this guide came from ISO 11607. The ASTM guide has formalized the aging process just short of specifying a test protocol. The accelerating aging test has many uncertainties and the ASTM committee did not have the necessary knowledge or the political will to establish a specific test standard. The FDA requires evidence that supports a manufacturer's claim regarding a product's shelf life. This requirement, as well as market demands, dictates the need for an accelerated aging program. Ultimately, these pressures make it very tempting for engineers to put better judgment aside and push the envelope of the Q_{10} theory in favor of shorter aging studies. (Swain, 2000)

Arrhenius Reaction Rate (Q_{10} Theory)

Medical devices and related packaging commonly use a Q_{10} coefficient of 2.0 in the accelerated aging factor formula. This is a conservative approach to determine the aging factor for polymeric systems in the moderate temperature range used by the medical device industry. The accepted range, according to ASTM F1980-99, is between 1.8 and 2.5; nevertheless, a reaction rate above 2.0 must be sufficiently characterized in the literature. More aggressive AAFs may be used with documented evidence to show a correlation between real-time and accelerated aging.

The ASTM F1980 guide states that the Arrhenius reaction rate function theory is directly related to packaging material compositions. It is based on the premise that the chemical reactions involved in the material degradation follow the Arrhenius theory, which was founded around a homogeneous process.

The ambient real-time room temperature (T_{RT}) used in the aging formula is between 20°C and 25°C. This is based on normal hospital type storage conditions. For example, if the real-time aging samples were stored in ambient conditions of 23°C then the T_{RT} should reflect the same value. A temperature of 25°C is the recommended conservative room temperature to utilize when detailed information about the storage environment is unknown. (ASTM, 1999)

Accelerated Aging Temperature (T_{AA})

One uncertainty of the Q_{10} theory is determining the appropriate accelerated aging factor (AAF), which is the ratio of the time necessary to achieve the same material property degradation as a package stored at real-time conditions. The greater the AAF value, the shorter the test time (Accelerated Aging Time = desired Real-Time / AAF). Engineers are pushing the conservative approach aside and raising the T_{AA} to appease market demands, therefore, reducing the time necessary to conduct a test. Some manufacturers are using temperature inputs of 75°C, which raises serious questions to the reliability of the test results. The ASTM guide strongly recommends not exceeding 60°C. In a presentation given by the chairperson of ASTM F1980-99, he was noted to strongly discourage exceeding a T_{AA} of 55°C. (Nolan, 2000)

The specified aging temperature needs to be carefully considered and chosen based on the characteristics of the material under investigation. As the test temperature increases, above industry standard levels, the effect of the high temperature on the material properties of the package system needs to be understood. The benefits of shortening the test duration needs to be balanced with the risks involved. Accelerated aging tests must be able to extrapolate the high temperature input properties to real-time room temperature properties in order to be valid.

Humidity

The focus of this research is on the effects of humidity and to understand it's relevance in the accelerated aging process. When this research began, the majority of accelerated aging tests conducted at Westpak Inc., a packaging and product testing lab in San Jose, California, incorporated humidity. Humidity is not a component of the Q_{10} theory. It is interesting to point out that humidity is not mentioned in the ASTM F1980 guide until the last page of the appendix (subtitled Non-mandatory Information). The guide notes that it may be necessary to consider the effects of humidity in conjunction with temperature.

As the temperature increases the percentage of relative humidity is often misunderstood. Absolute humidity is a measure of the amount of moisture in the air; weight of water vapor per unit volume or air (typically grains of water vapor in air). The grains of water vapor in the air at 65°C, 75% relative humidity (RH) are approximately double the grains of water vapor at 45°C, 75% RH. If ambient real-time storage conditions of 23°C, 50% RH are physically increased in a sealed test chamber to 65°C, the relative humidity would be less than 10%. However, the suspended grains of water vapor in the air would remain constant; as temperature increases the capacity of air can hold more water vapor.

The overall interpretation is that humidity is an additional stress factor that is not a component of the aging study. The reality is that a large percentage of aging studies incorporate humidity, and the effects of this additional stress factor needs to be scrutinized. (ASTM, 1999) Based on experience at the Westpak, Inc. testing laboratory, trends on controlling a high humidity condition are decreasing over time with the adoption of ASTM F1980.

Post Aging Test

After the aging test has been conducted, the physical properties and integrity of the material must be evaluated. For the purposes of this study the seal strength properties were examined. The ASTM guide recommends a comparison of the mechanical properties of the aged package materials to the samples stored for the actual real-time in a warehouse. Common industry practice of evaluating post-aged package systems focuses on the material and whole package integrity test. This study utilized the common industry practice of comparing peel-strength values. Suggested test methods that challenge the package material characteristics are seal strength, specifically ASTM F88. The accepted criteria for the minimum peel strength are based on the zero-time and real-time data. All of the package material test results were compared to the zero-time and real-time control samples. (ASTM, 1999)

CHAPTER 2

DEVELOPING TEST METHODOLOGY

ASTM F1980-99

Three-sided sealed pouches, measuring 12 x 5 inches, were utilized for this research (Refer to Figure 2.1). The pouches were visually examined for any obvious defects in accordance with ASTM D1886.

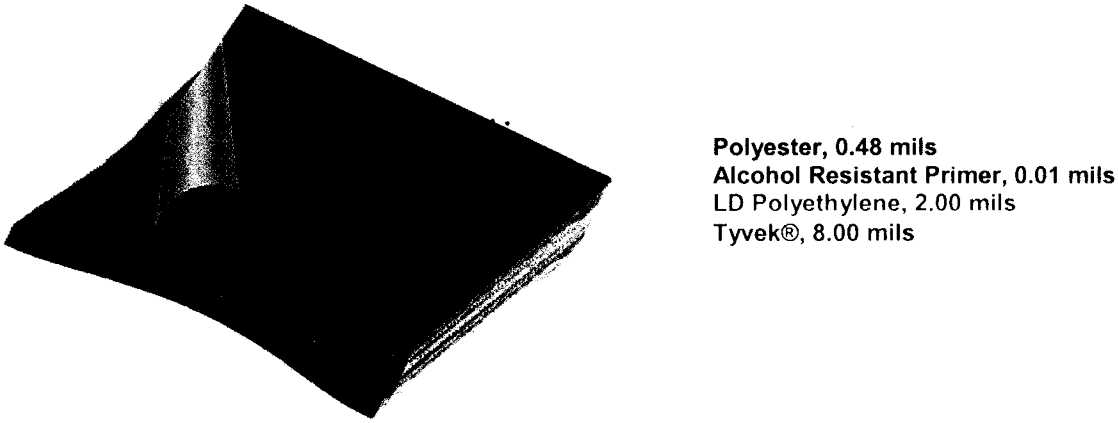


Figure 2.1 Lamination diagram

Next, a seven inch wooden dowel (in order to simulate a medical device catheter) was placed in each pouch and the ends were heat sealed. The pouches were divided into test groups with specific test durations according to table 2.1 (following page) and labeled with a permanent marker. The accelerated aging durations were calculated using the Q_{10} formula as outlined in the ASTM F1980 standard:

Figure 2.2 Sealed pouch

AAF = Q_{10} $[T_{AA} - T_{RT}/10]$
Accelerated Aging Time = RT (days)/ AAF

Where:

AAF = Accelerated Aging Factor
T_{AA} = Accelerated Aging Temperature
Q_{10} = 2.0 (conservative coefficient for polymeric systems)
T_{RT} = Ambient Real-Time Storage Temperature
RT = Real-Time Shelf Life

Example for one year aging of the 45°C samples (ambient real-time temperature 23°C)

$AAF = 2.0^{[45-23/10]}$
$AAF = 2.0^{[2.2]} \Rightarrow 4.595$

Accelerated Aging Time = 365 (days)/ 4.595 ⇒ **80** (days)

QTY OF POUCHES	TEST GROUP	TEST DURATION (days)
	INITIAL CONTROL SAMPLES	
10	Ambient	Zero Time
	ONE YEAR SAMPLES	
10	45°C Temperature Only	80
10	45°C Temperature @ 75 % Relative Humidity	80
10	55°C Temperature Only	40
10	55°C Temperature @ 75 % Relative Humidity	40
10	65°C Temperature Only	20
10	65°C Temperature @ 75 % Relative Humidity	20
	TWO YEAR SAMPLES	
10	45°C Temperature Only	159*
10	45°C Temperature @ 75 % Relative Humidity	159*
10	55°C Temperature Only	80
10	55°C Temperature @ 75 % Relative Humidity	80
10	65°C Temperature Only	40
10	65°C Temperature @ 75 % Relative Humidity	40
	REAL-TIME CONTROL SAMPLES	
5	65°C Control	20
5	55°C Control	40
5	45°C Control	80
10	3.5 Year @ Ambient Conditions	1278
*Note: samples were tested beyond their end test date; an additional 3 years of storage at ambient conditions		

Table 2.1 Accelerated aging and material test plan

The pouches were placed in their respective accelerated aging test chambers for the calculated test durations (Refer to Table 2.1). Following the conclusion of the aging test the pouches were removed from the test chamber and acclimated at ambient conditions for a minimum of 48 hours. Laboratory equilibrium was conducted in accordance with the general requirements of ASTM D4332. Next, the test specimens were subjected to the peel strength test in accordance with standard practices of the medical device industry.

Peel Strength Test Procedure

The original manufacturer's heat seals were subjected to the ASTM F88-94 "Seal Strength of Flexible Barrier Materials" peel test. Specimens were cut approximately 3.5 inches long x 1.0 inch wide in accordance with the test standard. The samples were pulled apart at an industry standard crosshead grip separation rate of 10 ipm (Refer to Figure 2.3). According to the ASTM F88 test procedure the grip separation rate cannot be less than 10 ipm and should be between 10 to 12 ipm. The samples were positioned and secured in the grips with the Tyvek® film located in the lower, fixed, jaw and the poly film positioned in the top-jaw.

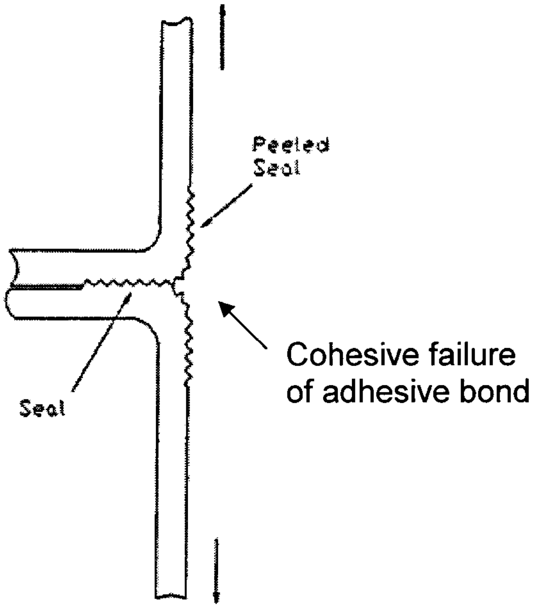


Figure 2.3 Specimen failure mode

Test Equipment

Instron, Chillton, and Shimadzu universal test machines were used to determine all of the material characteristics of the pouch package systems. Test results were captured and recorded on a Hewlett-Packard x-y plotter and Trapizium data acquisition software in the form of force versus deflection plots. The mean propagation force was derived manually by taking the faired value of the force versus deflection plot (between 20% and 80% region). The manual calculation was collected for the data recorded on the HP plotter. The data captured on Trapizium software were calculated using an automatic calculation for the Mean Peak Value (of the propagation region). Westpak, Inc. accelerated aging test chambers were utilized for the shelf-life studies.

The data from the 45°C 2-year and 3-year real-time periods were tested and evaluated on different pieces of equipment due to laboratory availability. The Shimadzu universal test machine utilized a digital electronic data acquisition system as apposed to the analog system used originally.

Limitations of Study

Due to time constraints and equipment availability, this study will not compare the accelerated aging samples to the real-life control samples at the 1 and 2-year time intervals. Real-life control samples were set aside and stored for 3.5-years before resources were available to conduct an evaluation. In addition, the 2-year designated 45°C real-time samples were not tested at the end of their 160-day duration due to issues beyond the control of the researcher. This included an additional three years of ambient storage.

The addition of the ambient storage makes the 45°C 2-year and 3.5-year real-time samples directly comparable. The practice of combining ambient storage with accelerated aging time designations is an accepted practice in the medical device industry. The 45°C 2-year and real-time samples were tested together when test facility and resources were available to complete the research. The long time span adds a minor level of complexity when comparing the data groups. Nevertheless, the long time span was a benefit to this research; the most significant variation in the data only occurred after 3.5-years of real-time aging, which would not have been discovered with the original design of experiment.

Comparison of the 45°C 2-year and real-time samples were collected on a digital acquisition system with a data rate collected at 50 msec. The initial data (all preceding groups) were captured and recorded on an analog system with a pen plotter. Common industry practice compares data collected from either system without any issues.

This research utilizes six controlled accelerated aging test chambers for considerable lengths of time. Access to these chambers was a limiting factor due to the range of temperature and humidity variables for this research. Validation of the end seal on these pouches was beyond the scope of this research. The end seal was created in an uncontrolled process, used to seal the dummy device and prevent air transfer within the pouch (simulating real world conditions). The supplier of the pouches validated the three manufacturer side seals, which were used for this study. Therefore, sterile integrity testing was not conducted due to the lack of consistent end seals.

CHAPTER 3

JOURNAL ARTICLE

This article is to be submitted to the

Pharmaceutical & Medical Packaging News Journal

and, therefore, has a different format from that of Chapters 1, 2, and 4.

EFFECT OF HUMIDITY ON ACCELERATED AGING OF MEDICAL DEVICE POUCHES

James R. Wise

May 2004

ABSTRACT

Tyvek®/polyester-polyethylene laminate pouches with water based adhesive were subjected to an accelerated aging test at the temperature levels of 45°C, 55°C, and 65°C (ambient humidity) for 1 and 2-year shelf lives. In addition, the accelerated aging tests were conducted at these three temperature levels simultaneously at elevated humidity conditions of 75% RH. The purpose of this research was to identify the effect of humidity in the accelerated aging process of medical device pouch package systems.

The data resulted in no significant difference from the high humidity test input. The test results showed that time was the leading factor in the degradation of the medical device pouch heat seals evaluated. In addition, the findings showed that the current industry practice of only recording peak force may be an over estimate of the seal strength. The mean propagation force was noted to give a truer representation of the heat seal characteristics.

INTRODUCTION

The focus of this research is to determine the degradation effects that humidity has on the seal strength characteristics of a four-side sealed Tyvek®/polyester-polyethylene laminate pouch. The effects of the temperature and humidity on the package system need to be accounted for.

The medical device industry faces many packaging challenges; their products are intended for hospital operating room environments and are used as surgical tools or implants. The package system must be able to survive the sterilization process, distribution cycle, storage, and ultimately introduce a sterile medical device to a surgical team. Any compromise to the sterile integrity of the package system is not acceptable and places human life and public safety at immediate risk.

The FDA requires that a shelf life must be declared for a medical device package system. This requirement is accomplished by conducting real-time shelf life testing. In order to ensure public safety and well being, a process was created to speed the innovation of medical technology to the market. Accelerated aging studies subject the package to elevated temperature and time inputs in order to simulate real-life aging. The theory is based on the Arrhenius Equation where increased temperatures accelerate the molecular activity of materials. Industry consensus test standards recommend procedures and methods for accelerated aging studies that meet the medical device requirements.

BACKGROUND

ASTM F1980-99

Tyvek®/polyester-polyethylene laminate pouches with water based adhesive were utilized for this research. In order to simulate a medical device catheter, seven inch wooden dowels were placed in each pouch and the ends were heat sealed. The pouches were divided into test groups at the temperature levels of 45°C, 55°C, and 65°C (ambient humidity) for 1 and 2-year shelf lives. In addition, the accelerated aging tests were conducted at these three temperature levels simultaneously at elevated humidity conditions of 75% RH.

The accelerated aging durations were calculated using the Q_{10} formula as outlined in the ASTM F1980 test standard. The recommended Q_{10} coefficient value of 2.0, as well as, an ambient real-time temperature of 23°C was utilized. These test variables can be adjusted slightly, which may result in a significant change to the overall test duration. Conservative values were used to minimize test variability and ensure credibility. The accelerated aging calculations used a consistent Q_{10} coefficient and ambient temperature for all three aging test temperatures.

Temperature and Ambient Humidity 1 and 2-Year test inputs	Temperature and High Humidity 1 and 2-Year test inputs
45°C @ <10%	45°C @ 75%
55°C @ <10%	55°C@ 75%
65°C @ <10%	65°C@ 75%

Note: Control samples were conditioned at ambient laboratory temperature and humidity levels.

Table 3.1 Accelerated aging test chambers

Test Procedure

The pouches were placed in their respective accelerated aging test chambers for the calculated one and two year test durations. Following the conclusion of the aging test, the pouches were removed from the test chamber and brought to equilibrium at ambient conditions for a minimum of 48 hours. Next, the samples were subjected to the peel strength test in accordance with standard practices of the medical device industry.

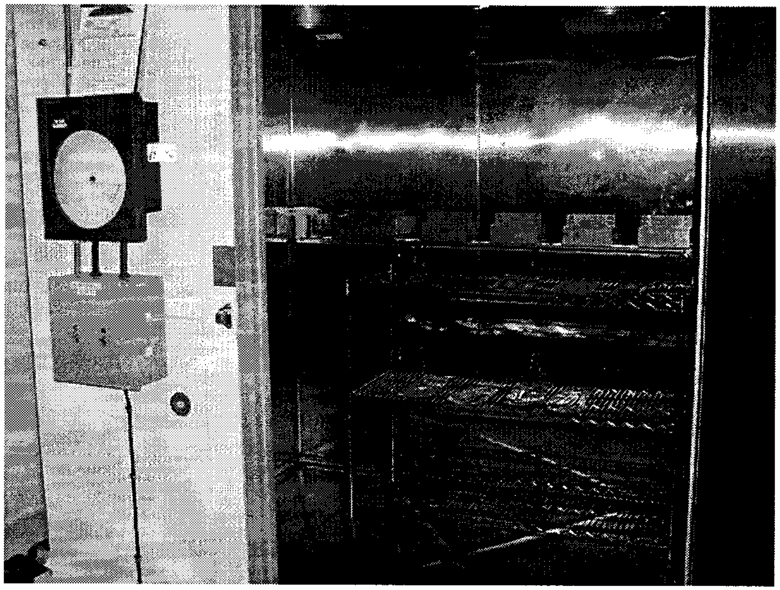


Figure 3.1 Test chamber

The original manufacturer's side heat seals were subjected to ASTM F88-94 "Seal Strength of Flexible Barrier Materials" peel test. The samples were cut into approximately 3.5 inches long x 1.0 inch wide specimens in accordance with the test standard. The samples were positioned in the grips with the Tyvek® film located in the lower, fixed, jaw and the poly film positioned in the top-jaw. The samples were pulled apart at an industry standard crosshead grip separation rate of 10 ipm. The industry standard peak force required to peel apart the seal was collected on an instrumentation system. In addition, the mean propagation value of the center section of the force versus deflection plot was also recorded (not a current common industry practice).

Figure 3.2 Test fixture

The force versus deflection plot is generally viewed as two distinct sections. The first section, referred as the initial *force,* is the force required to break the bond and start the peeling process (identified as the seal resists the tensile load applied from the test apparatus). The second section, known as the *propagation force*, is the response data as the width of the heat seal fails (identified as the plateau section of the plot). Refer to the following diagram, Figure 3.3

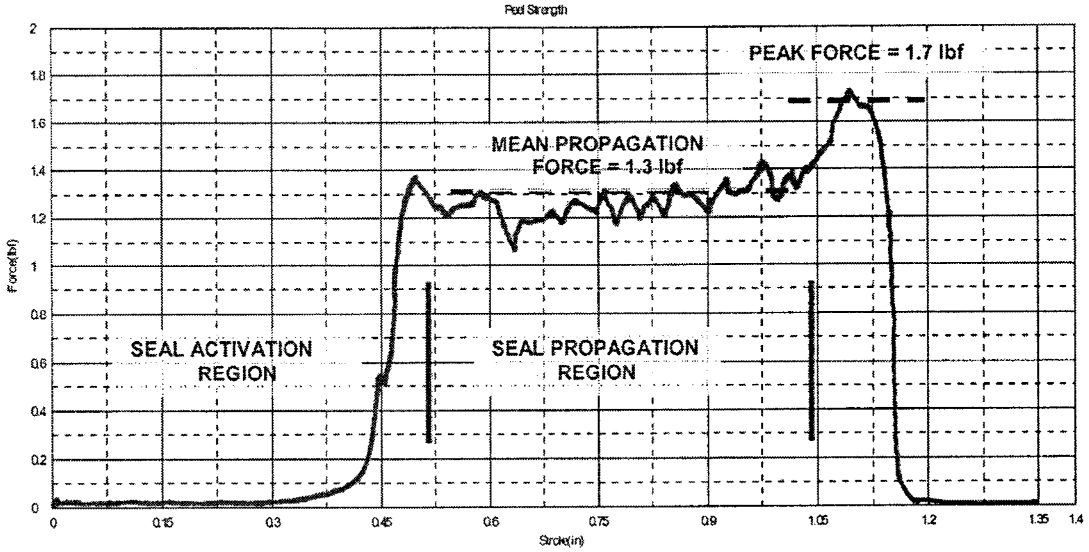


Figure 3.3 Force versus deflection plot

RESULTS

Peel Strength

The peel test subjected one inch wide samples to a separation test at a jaw crosshead rate of 10 ipm. Refer to Figures 3.4 through 3.11 for the peak and mean propagation data collected, including the average bar (dashed lines) for each data set. The standard deviation was noted to be significantly lower on the mean propagation data versus the peak force (noted in the legend of each chart). The mean propagation force was noted to be very consistent when compared to the peak force values.

65°C Accelerated Shelf Life

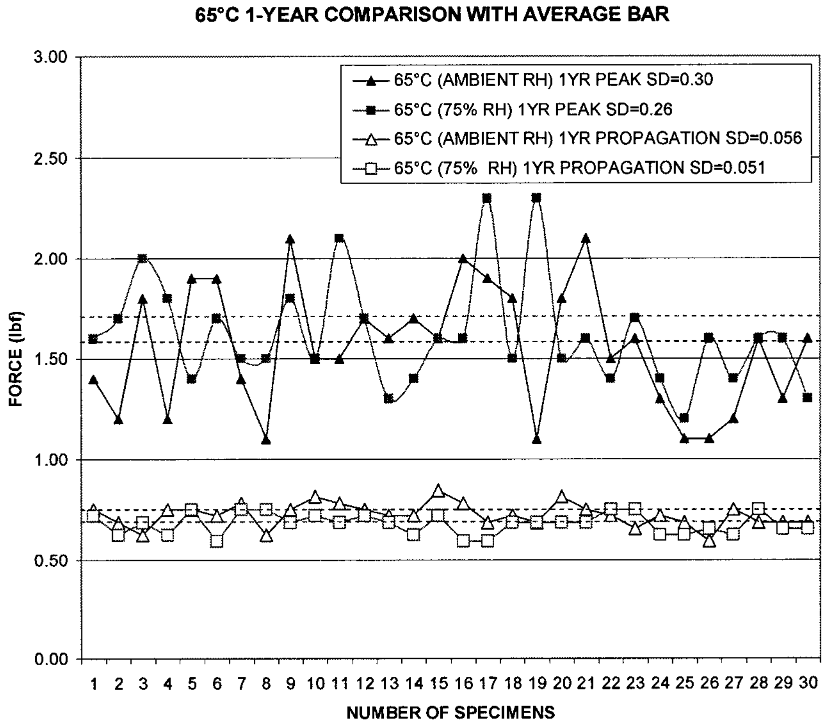


Figure 3.4 65°C 1-year data set

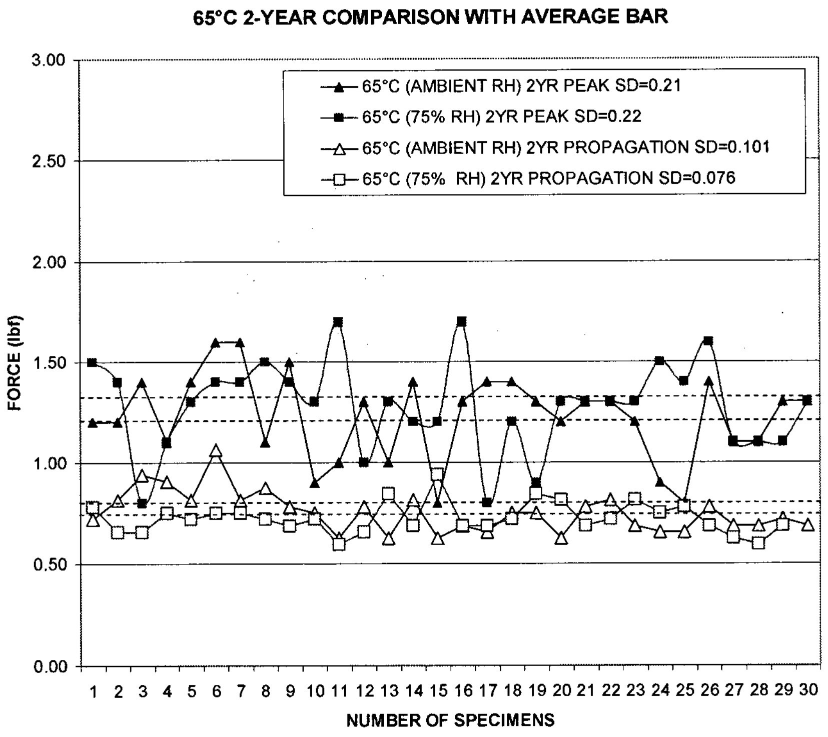


Figure 3.5 65°C 2-year data set

Figures 3.4 through 3.6 display the average peak force of the peel strength to be noticeably higher on the specimens subjected to the 75% humidity conditions (65°C & 55°C 1-yr). Examination of the same figures shows the reverse trend for the mean propagation force. The 75% humidity condition showed a lower value than the ambient RH data set. Further testing on samples that were subjected to aging for a duration of 80 days (in a temperature and humidity chamber) shows similar conflicting results (Refer to Figures 3.7 and 3.8).

55°C Accelerated Shelf Life

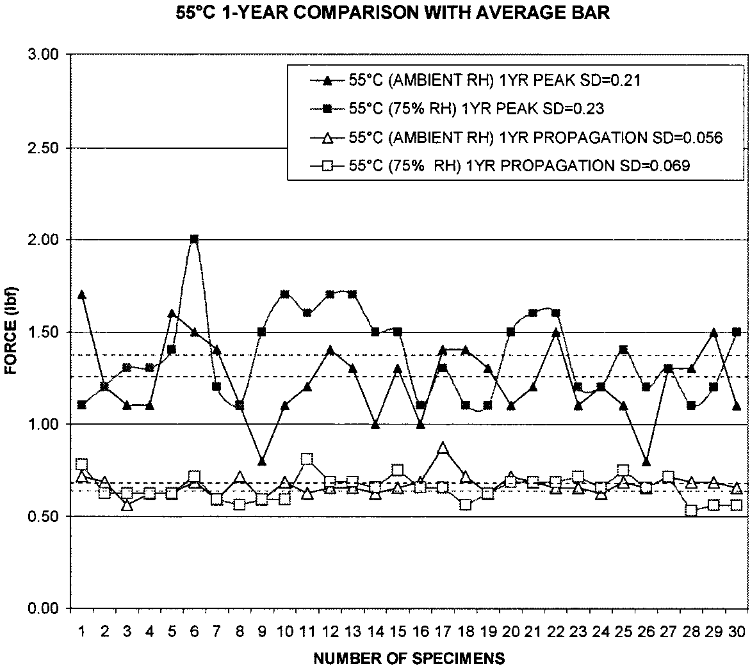


Figure 3.6 55°C 1-year data set

The 55°C 1-year data (Refer to Figure 3.6) was noted to have consistent results for the mean propagation force. The average results were 0.67 lbf ambient and 0.66 lbf 75% RH. The difference of 0.01 lbf is very minor and does not show a trend in this data, it is within 1-sigma standard deviation of 0.056. The lack of a clear trend for the high humidity results was noted for all data sets collected. The values for 75% were not consistently higher or lower than the ambient samples. The peak force showed a higher level of variation, which was typical of the entire data collection in this research. For all of the data collected, the average peak force ranged between 1.24 lbf and 1.99 lbf with a standard deviation range of 0.18 to 0.39.

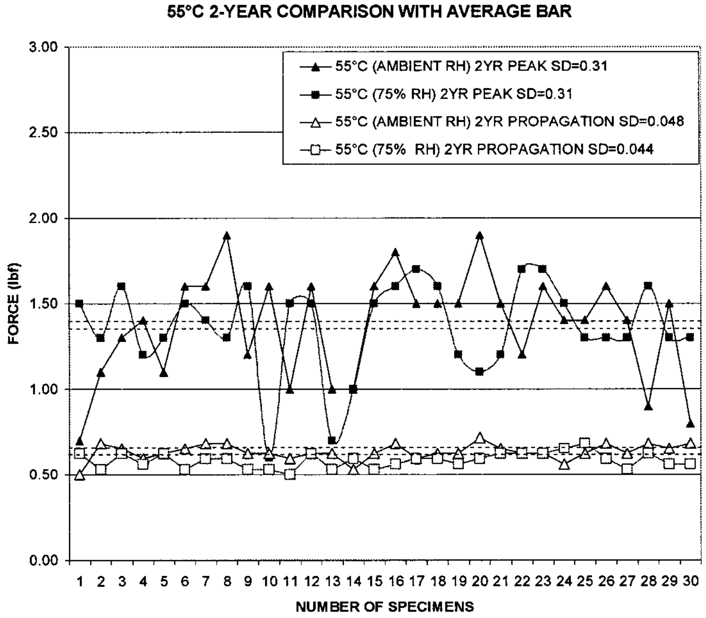


Figure 3.7 55°C 2-year data set

45°C Accelerated Shelf Life

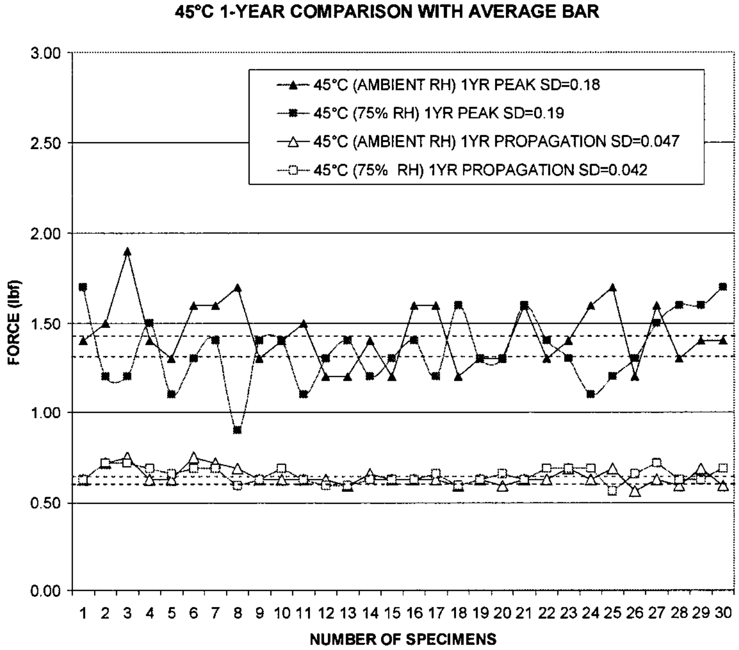


Figure 3.8 45°C 1-year data set

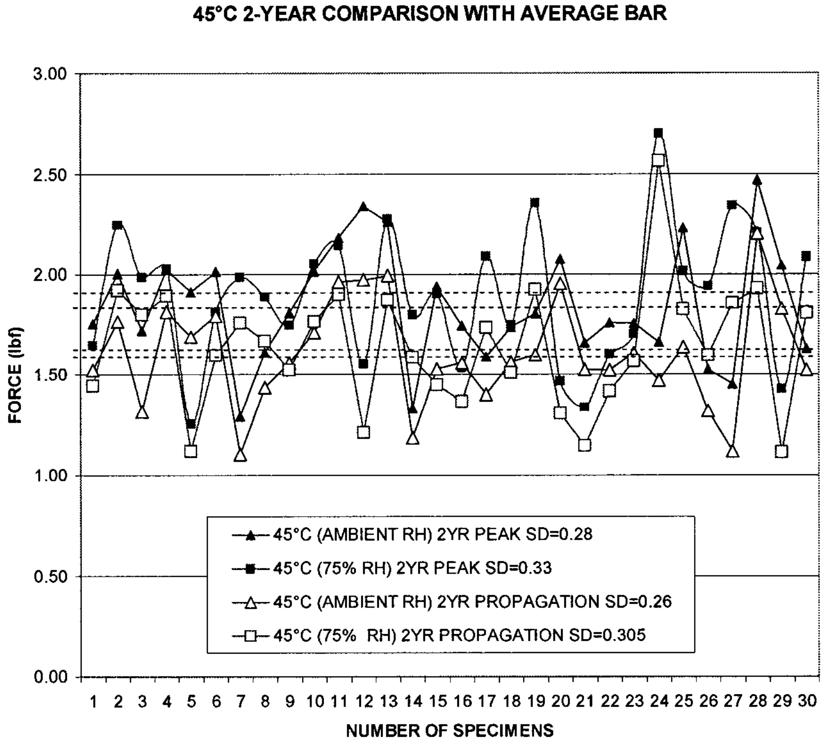


Figure 3.9 45°C 2-year data set

The 45°C 2-year data was noted to have a significantly higher average mean propagation force. This was consistent for both the ambient and 75% humidity groups. The mean propagation values for the preceding groups were approximately 0.70 lbf. The data collected after the long-term duration increased to approximately 1.60 lbf. This increase was noted to be a doubling of the mean propagation force. The doubling of the mean propagation results was also evident on the 3.5-year real-time data set. This type of dramatic increase in the separation force was not observed in any other group. The peak force did not show similar results; the increase was noted to be from approximately 1.44 lbf to 1.90lbf.

Control and Real-Time

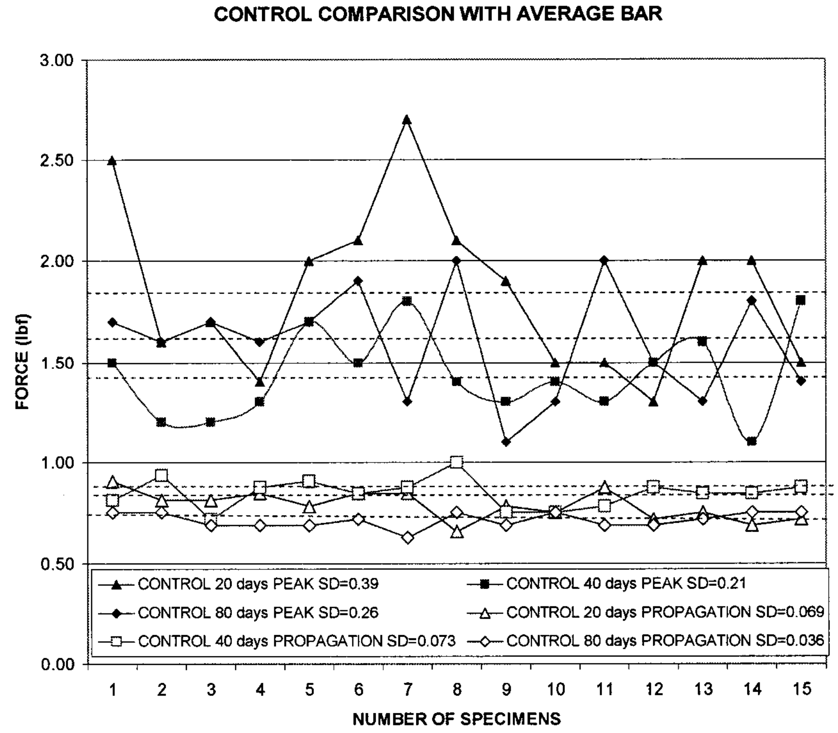


Figure 3.10 Control data set

The control samples were noted to be higher in both the peak and mean propagation forces than their corresponding aging groups (Refer to Figure 3.7). The 20 day control group corresponds to the 65°C 1-year, the 40 day control group corresponds to the 65°C 2-year and 55°C 1-year, and the 80 day control group corresponds to the 55°C 2-year and 45°C 1-year. The higher values ranged between 1.44 to 1.85 lbf for the peak force and 0.71 to 0.85 lbf for the mean propagation levels.

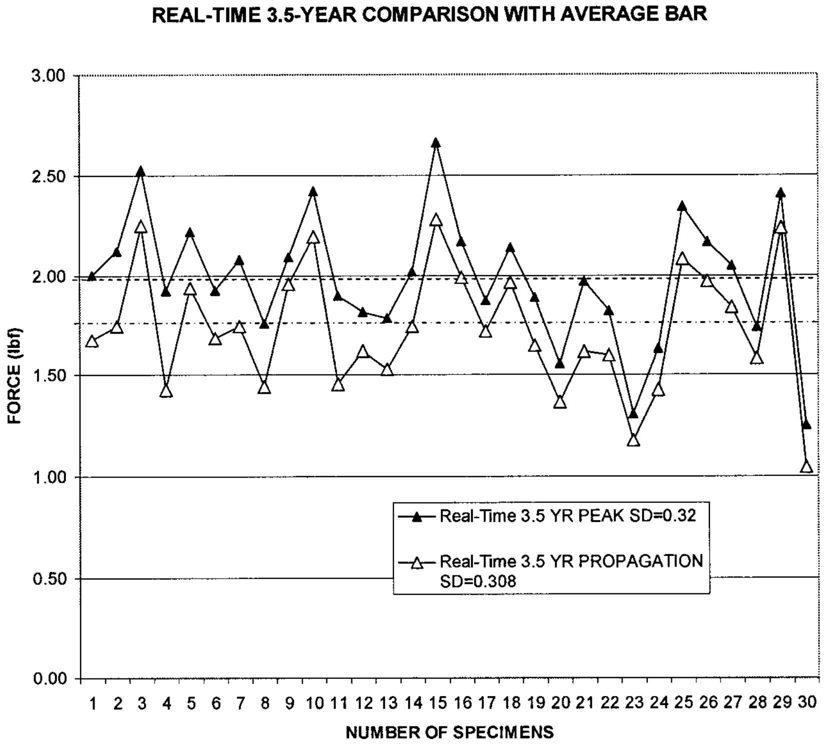


Figure 3.11 3.5-Year real-time data set

The 3.5-year real-time aging group showed a similar trend (Refer to Figure 3.11). The peak and mean propagation values were noted to be slightly higher than the groups subjected to elevated temperature and humidity accelerated aging inputs.

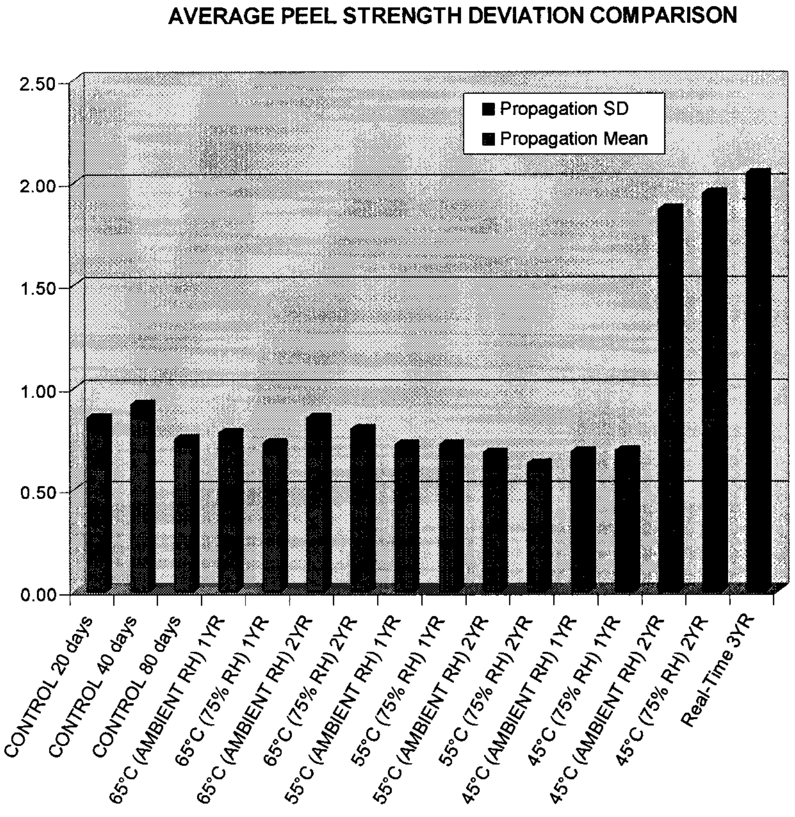


Figure 3.12 Overall data

Based on the test results, it is not relevant to use the peak force to analysis the data from this point forward. The chart in Figure 3.12 shows a major finding, change over time tracked with the mean propagation values. The long-term data (45°C 2-yr & 3.5-yr RT) were noted to have higher peel separation forces and standard deviations. The mean propagation data set was noted to have the values increase over time irrespective of the temperature or humidity inputs.

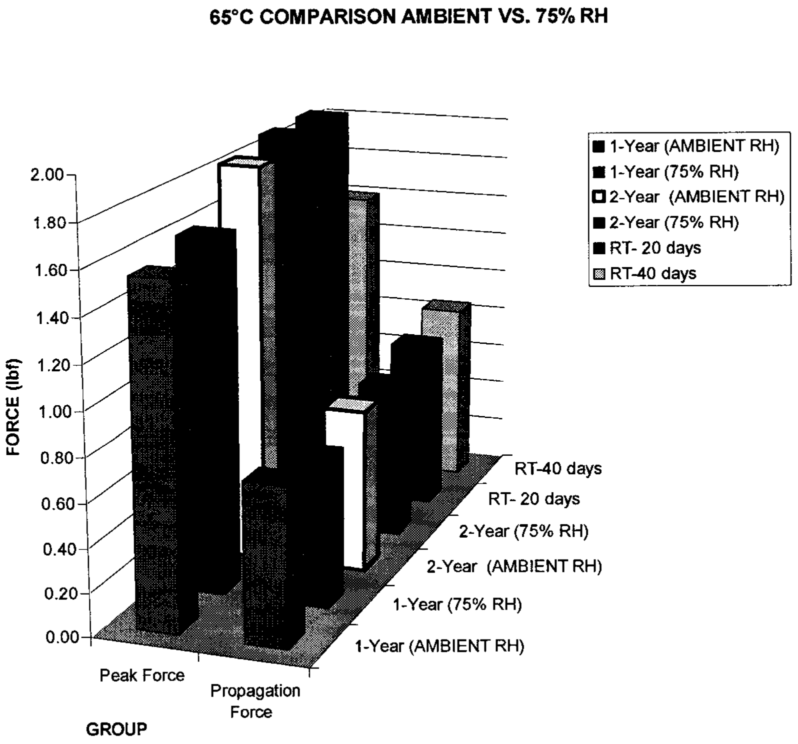


Figure 3.13 65°C ambient versus 75% RH

Figures 3.13 through 3.15 compare only the affect of humidity to the specific temperature group. These graphs show that the humidity input, when viewed independently, does not influence the heat seal separation force. The data show that the temperature and time inputs have the greatest influence over the package characteristics.

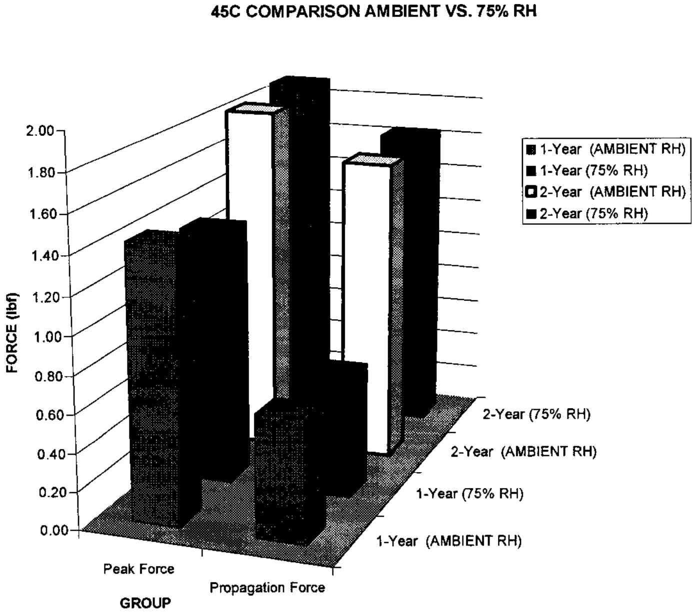


Figure 3.14 45°C ambient versus 75% RH

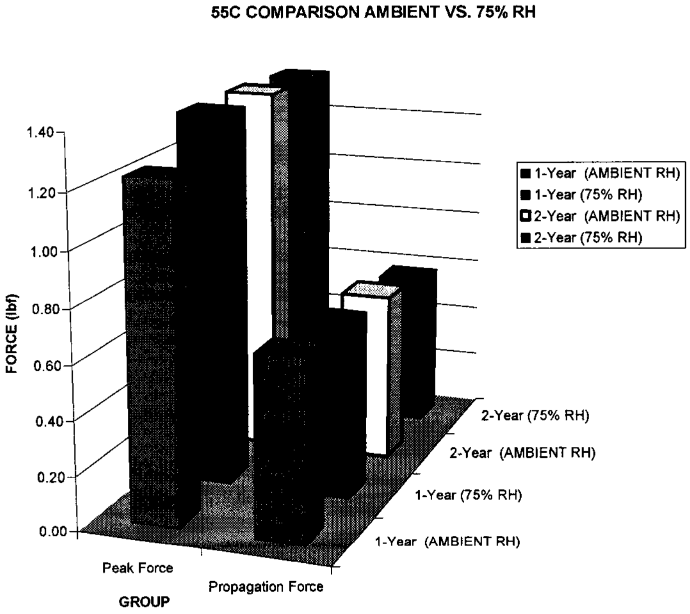
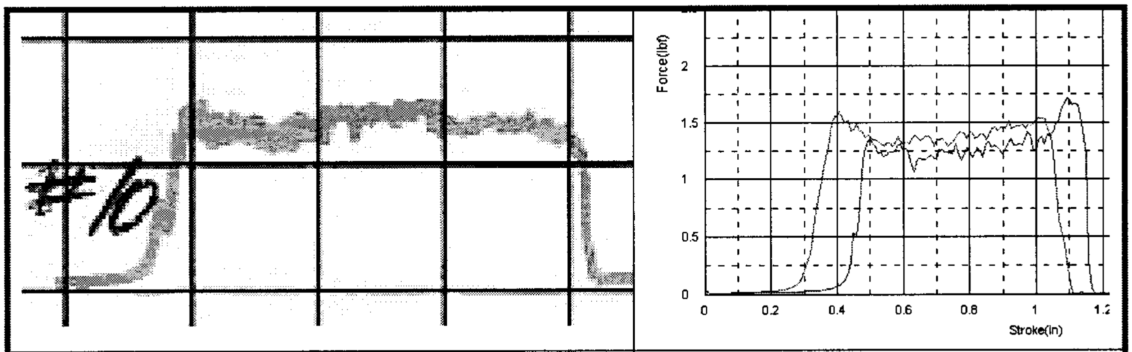


Figure 3.15 55°C ambient versus 75% RH

It is important to note that the initial and long-term data were captured and recorded on different pieces of test equipment. The initial data (65°C, 55°C, and 45°C 1-year) were collected on an Instron test machine with an analog signal and pen plotter. The long-term data (45°C 2-year and real-time) were collected on a Shimadzu system with a digital readout. An evaluation was conducted to verify the validity of the data collected on the Shimadzu system. A sample group of long-term data was tested on the Instron analog equipment; the force versus deflection profiles and values were consistent. It was determined that the long-term data were valid and that the use of different test equipment had no adverse effects on this research (Refer to Figure 3.16). This data reinforces the conclusion that the dramatic change in the results of the long-term data were attributed to time.



45°C Ambient 2-Year Sample (1.25 lbf per division) Instron Equipment

45°C Ambient 2-Year Shimadzu Equipment

Figure 3.16 Equipment validation on same time group

The data are inconsistent and do not show clear trends that a high humidity environment has an affect on the peel strength of medical device pouches. In many cases the peak force was higher for the high humidity group, but the mean propagation force was lower. The separation force values oscillated back and forth randomly for the groups with and without high humidity. No distinct trend was interpreted that showed that humidity played a critical role in the accelerated aging process.

This research found that the use of the peak force collected during the peel test was very inconsistent. The mean propagation force has a lower range of variation in the data with a lower standard deviation. The mean propagation force requires sophisticated data collection software to calculate and acquire the value automatically, but results in an improved understanding of the material characteristics. Manually collecting the mean propagation force is cumbersome and less accurate. Use of the current method (peak force) did not provide any meaningful interpretations of the test results. The mean propagation force (higher than industry standards) highlighted a clearer picture of the pouch peel strength characteristics. Large variations of the peak separation force were noted within the sample population. Thirty specimens were tested for each sample group. The standard deviation was noted to be slightly above 0.20 for a majority of the sample groups. Comparing the data from one time and temperature group shows that the difference was within 1-sigma standard deviation; statistically insignificant. This is inherent with this type of test and it is consistent with the manufacturer's initial peel strength results.

Very minor variations were noted for the initial data groups across the board, with the exception of the 45°C 2-year and 3.5-year real-time data. The variation for the mean propagation force was noted to range between 0.58 and 0.79 lbf. The range of 0.21 lbf was not considered a major difference for all of the groups evaluated; no trends were noted while interpreting this data.

The data captured and recorded for the long-term groups show a significant increase in the center section of the force versus deflection plot. The center region, discussed as the propagation force, is directly related to the cross section of the heat seal. The increased force in this area was consistent for both the 45°C 2-year and 3.5-year real-time aging groups.

The signature of the force versus deflection plots changed after the 45°C 2-year samples, including the real-time data. The initial data show a relatively low propagation force of ~0.8 lbf and a standard peak force level of ~2.0 lbf. The industry standard method of qualifying the data is to capture the peak value only. The initial data show a spike in the force as the peel reached the outside of the seal. This spike is likely due to an uneven heat distribution along the width of the sealing bar. The seal has greater fusion on the outside due to less thermal mass of the package system as opposed to the inside seal edge. The result is more heat transfer to the outside seal creating the large spike in the profile.

A statistical t-test was conducted on the initial peak force data (65°C, 55°C 1-yr and 2-yr, and 45°C 1-yr) in order to determine the confidence level of comparing two populations. This t-test was used to compare the high and low humidity results for the specific temperature/time categories defined in tables 4 and 8. This test showed that the 55°C and 45°C 1-year samples had the greatest confidence level of approximately 97.5 to 99%. These sample groups did not show consistent results when compared to one another individually; that is to say that the effect of humidity is not significant. The remaining groups had confidence levels below 90%. A confidence level below 90% was determined as the cut off point for determining reliability of the results. A confidence level of below 90% is considered to have no significant difference between the two populations being compared. The t-test reinforces the interpretation of the data that humidity has no significant influence over the peel strength results.

Using industry standard techniques, the actual peel strength is misrepresented as the spike in the profile. Based on experience of peel testing conducted at Westpak, Inc. testing laboratory, the spike is a common occurrence in the industry. The data collected in this report shows the spike consistently on the outside of the seal.

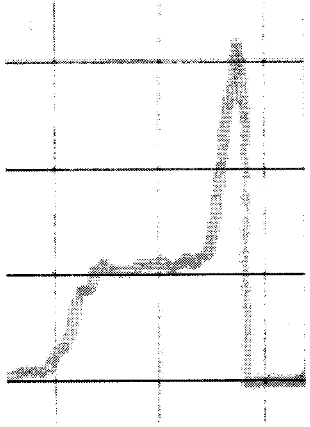
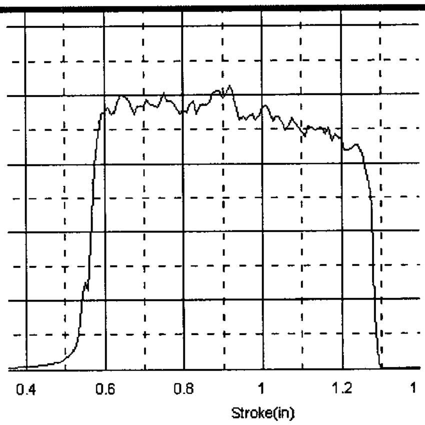
	
45°C Ambient 1-Year Sample (0.5 lbf per division) Instron Equipment	45°C Ambient 2-Year (0.5 lbf per major division) Shimadzu Equipment

Figure 3.17 Profile of force versus deflection plots same temperature group

The long-term data show that the force required for breaking the bond of the center section of the seal increased over time (Refer to Figure 3.17). This increase reduced the occurrence of the spikes in force at the outside edge of the seal. The increase in the propagation force was approximately equal to the spikes noted in the initial data collection.

The data show that the seal strength loses elastic properties and becomes more brittle; resulting in a higher propagation force. This test observation was noted to be irrespective of the temperature and humidity inputs. Prior to stress testing, all samples were stored at ambient laboratory conditions. The only test variable that was different between the initial and long-term data was time.

The data showed a minor trend of a reduction in the mean propagation force for the 1-year data. This trend is only evident with the data subjected to the simulated aging process (refer to Figure 3.18).

The control data was not consistent. The real-time data and 45°C 2-year data (with added real-time) showed that the force increased by a factor of two. There was no evidence of increased forces as a function of temperature or humidity test inputs. Therefore, the overriding contributing factor must be time. The increase and change of the profile was not thermally dependent, rather oxidation or chemically dependent. The increase was likely a curing of the adhesive coating of the Tyvek® film.

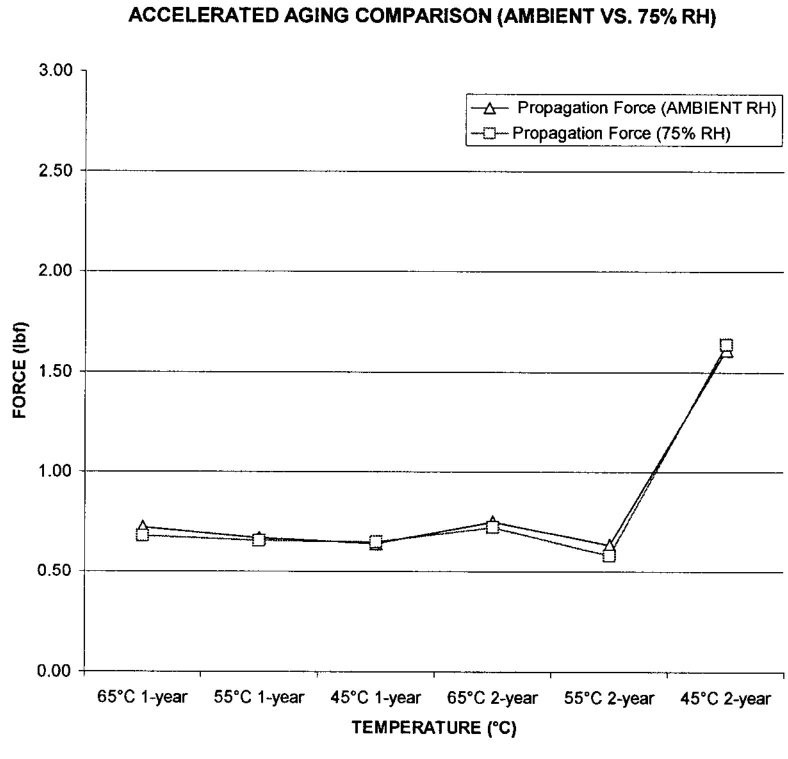


Figure 3.18 Accelerated aging comparison

CONCLUSION

The hypothesis of this research was that high humidity inputs did not have an effect on the shelf-life validation process of Tyvek®/polyester-polyethylene laminate pouches. The results of the peel strength tests show that added humidity stress input has no significant effect. The peel strength properties of the Tyvek®/polyester-polyethylene laminate pouch were not significantly altered by the introduction of a 75% relative humidity environment. The data show inconsistent results, leading to the conclusion that peel strength is not significantly altered when exposed to high humidity conditions.

Polyethylene and the Tyvek (spun bonded olefin) are non-polar materials, but water is a polar substance. Weaker electrical attractions than an atomic bond allow a molecule to develop temporary or permanent polarities that act to attract the opposite poles on a nearby molecule. The non-polar films in direct contact with the adhesive seal resist the adsorption of polar water. This acts a barrier for the moisture to penetrate and interact with the adhesive bond of the seal (Soroka, 1995).

The webs of the pouches formed a barrier for the adhesive coating; both materials were noted to have low water vapor transmission rates. This barrier property limited the adhesive from exposure to water vapor in the high humidity test chamber. Nevertheless, the effects of humidity need to be further studied on polar material lamination combinations typically used in the medical device industry.

The leading factor for significant changes to the peel strength values, during the shelf-life process, was time dependent. The real-time data were noted to change dramatically with a doubling of the mean propagation force over the course of 3.5 years. The high temperature inputs did not show any consistent trends for either increasing or reducing the force necessary to separate the peel-seal.

The results show that the ultimate validation must be centered on the real-time data. Medical device manufacturers should incrementally test their real-time sample every 6 months in order to declare a long-term shelf-life. This will allow the device manufacturer to observe the degradation of their package system over time, ensuring that the sterile integrity of the seal package systems remains intact.

Another significant finding was that the practice of analyzing the data based on the peak force could be misleading. Spikes were noted in the force versus deflection profile that were typically double the mean propagation force. Good laboratory practices should report the mean propagation force when analyzing heat seal separation data.

ACKNOWLEDGEMENTS

The author would like to thank Westpak, Inc. of San Jose and Oceanside, California, for the use of their testing equipment and facilities. Additional thanks are extended to Tolas Health Care Packaging of Los Angeles, California, for providing the pouch material used in this study.

REFERENCES

American Society for Testing and Materials. (1999) Selected ASTM Standards on Packaging. (4th ed.) Philadelphia, PA: Author

Brandenburg, R. K., & Lee, J. J. (1993). Fundamentals of Packaging Dynamics (5th ed.), Skaneateles, NY: L.A.B.

Marcondes, J., Schueneman, H. (1999). Engineering of Protective Packaging Class Notes. San Jose, CA: San Jose State University, Department of Nutrition and Food Science.

Nolan, P. (2000, January). ASTM Update: Methods Used to Evaluate Medical Device Package Integrity. Symposium conducted at the Medical Packaging Symposium, Anaheim, CA.

Swain, E. (2000, January). Putting standards to the test. Pharmaceutical & Medical Packaging News, 38-47.

Soroka, W. (1995). Fundamentals of Packaging Technology. Herndon, VA: Institute of Packaging Professionals.

CHAPTER 4

SUMMARY AND RECOMMENDATIONS

Summary

The hypothesis was stated that the humidity level would not have a measurable effect due to the added stress factor. The data are inconsistent and do not show clear trends that a high humidity environment has an affect on the peel strength of medical device pouches. No distinct trend was interpreted that showed that humidity played a critical role in the accelerated aging process.

This research found that the use of the peak force collected during the peel test was very inconsistent. The mean propagation force had a lower range of variation in the data with a lower standard deviation. The mean propagation force highlighted a clearer picture of the pouch characteristics.

A statistical t-test was conducted on the initial peak force data (65°C, 55°C 1-yr and 2-yr, and 45°C 1-yr) in order to determine the confidence level of comparing two populations. This t-test was used to compare the high and low humidity results for the specific temperature/time categories defined in tables 4 and 8. This test showed that the 55°C and 45°C 1-year samples had the greatest confidence level of approximately 97.5 to 99%. These sample groups did not show consistent results when compared to one another individually; that is to say that the effect of humidity is not significant. The remaining groups had confidence levels below 90%. A confidence level below 90% was determined as the cut off point for determining reliability of the results. A confidence level of below 90% is considered to have no significant difference between the two populations being compared. The t-test reinforces the interpretation of the data that humidity has no significant influence over the peel strength results.

The data captured and recorded for the long-term groups show a significant increase in the center section of the force versus deflection plot. The increased force in this area was consistent for both the 45°C 2-year and 3.5-year real-time aging groups.

The initial data show a spike in the force as the peel reached the outside of the seal. This spike is likely due to an uneven heat distribution along the width of the sealing bar. The seal has greater fusion on the outside due to less thermal mass of the package system as opposed to the inside seal edge. The result is more heat transfer to the outside seal creating the large spike in the profile.

The long-term data show that the force required for breaking the bond of the center section of the seal increased over time (Refer to Figure 3.13). This increase in the test response data reduced the occurrence of the spikes in force-pounds at the outside edge of the seal. The increase in the propagation force was approximately equal to the spikes noted in the initial data collection.

The data show that the seal strength loses elastic properties and becomes more brittle; resulting in a higher propagation force. The initial slope of the profile noted on Figure 3.16, shows that the modules of elasticity increase over time. The angle of the slope of the profile increased over time. This test observation was noted to be irrespective of the environmental temperature and humidity inputs.

The control data were not consistent. There was no evidence of increased forces as a function of temperature or humidity test inputs. Therefore, the overriding contributing factor must be time dependent.

Recommendations

The data show inconsistent results, leading to the conclusion that peel strength is not significantly altered when exposed to high humidity conditions. Nevertheless, the effects of humidity need to be further studied on other material lamination combinations typically used in the medical device industry. The data shows that accelerated aging tests have no added benefit to arbitrarily including humidity in the protocol. Accelerated aging tests are simpler and less costly to perform when ambient humidity conditions are specified; a benefit to the medical device industry.

In addition, the ultimate validation must be centered on the real-time data. Medical device manufacturers should incrementally test their real-time sample every 6 months in order to declare a long-term shelf-life. This will allow the device manufacturer to observe the degradation of their package system over time, ensuring that the sterile integrity of the seal package systems remains intact. All the data indicated that the real-time performed better than the accelerated aging group, with higher seal strengths. The accelerated aging tests were noted to have lower results than the 3-year real-time data and are considered to be a more conservative test.

Another significant finding was that the practice of analyzing the data based on the peak force could be misleading. Good laboratory practices should report the mean propagation force when analyzing heat seal separation data. This testing is extremely time intensive. Further research would benefit from conducting long-term real-time tests. Also, accelerated test conditions should correspond to the glass transition temperature (T_g) of the materials under evaluation.

REFERENCES

American Society for Testing and Materials. (1999) Selected ASTM Standards on Packaging. (4th ed.) Philadelphia, PA: Author

Brandenburg, R. K., & Lee, J. J. (1993). Fundamentals of Packaging Dynamics (5th ed.), Skaneateles, NY: L.A.B.

Marcondes, J., Schueneman, H. (1999). Engineering of Protective Packaging Class Notes. San Jose, CA: San Jose State University, Department of Nutrition and Food Science.

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Swain, E. (2000, January). Putting standards to the test. Pharmaceutical & Medical Packaging News, 38-47.

Soroka, W. (1995). Fundamentals of Packaging Technology. Herndon, VA: Institute of Packaging Professionals.

APPENDICES

Appendix A

Test Equipment

Figures A.1 displays the universal test equipment and sample fixturing utilized for this research

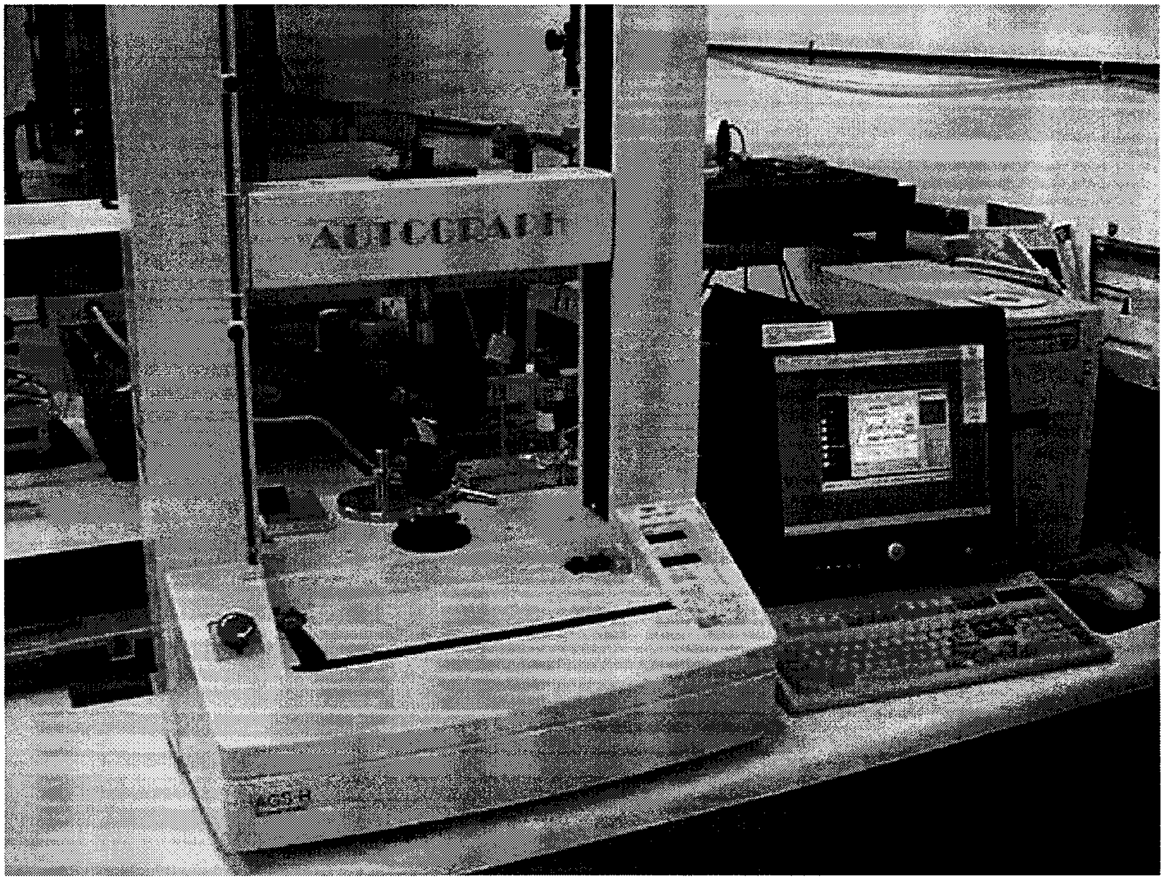
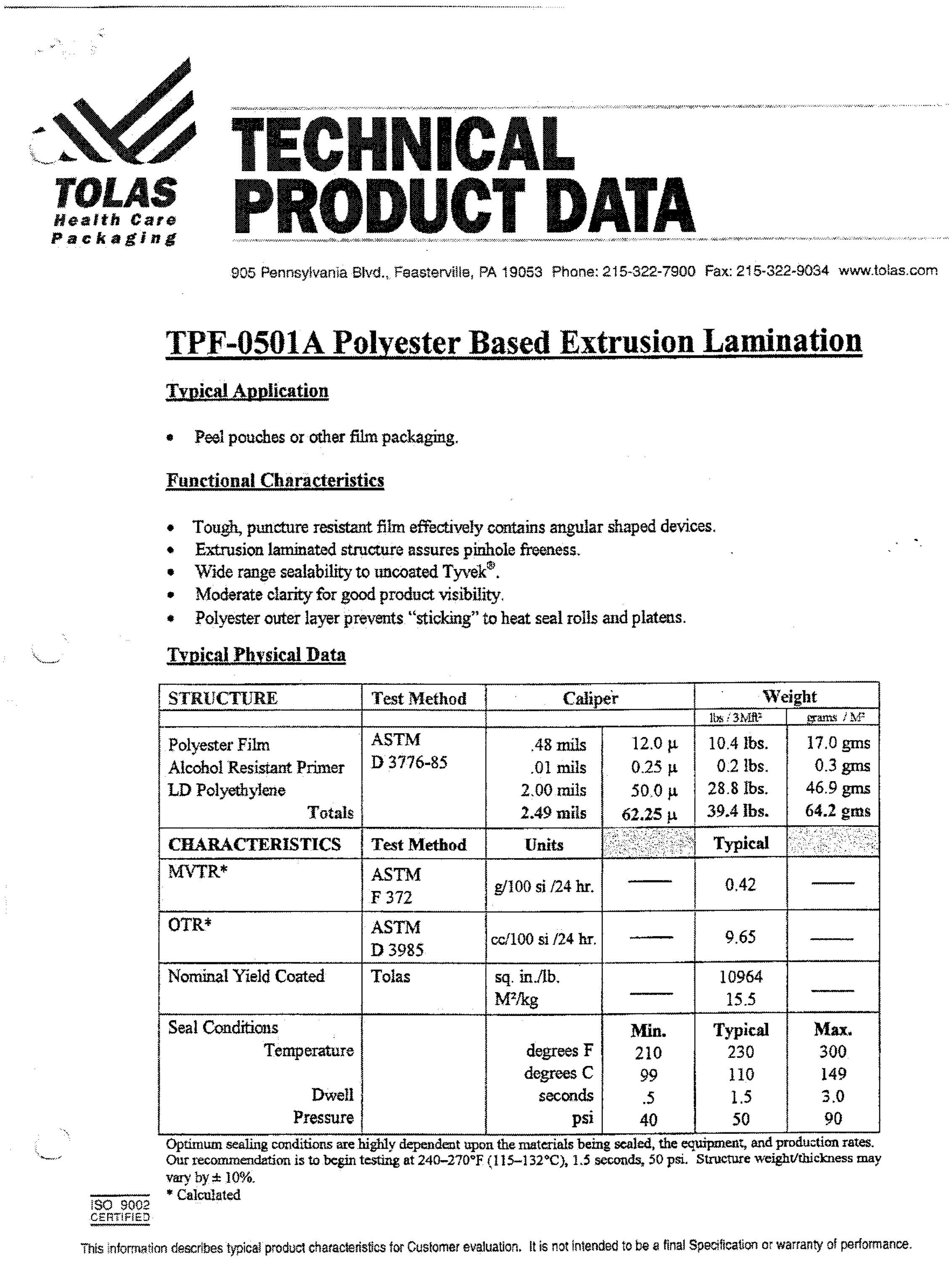


Figure A.1 Universal test Equipment

Appendix B

Material Specification Data

Figures B.1 through B.5 display the material specification data sheets supplied by the pouch manufacturer utilized in this research.



TOLAS
Health Care
Packaging

TECHNICAL PRODUCT DATA

905 Pennsylvania Blvd., Feasterville, PA 19053 Phone: 215-322-7900 Fax: 215-322-9034 www.tolas.com

TPF-0501A Polyester Based Extrusion Lamination

Typical Application

- Peel pouches or other film packaging.

Functional Characteristics

- Tough, puncture resistant film effectively contains angular shaped devices.
- Extrusion laminated structure assures pinhole freeness.
- Wide range sealability to uncoated Tyvek®.
- Moderate clarity for good product visibility.
- Polyester outer layer prevents "sticking" to heat seal rolls and platens.

Typical Physical Data

STRUCTURE	Test Method	Caliper		Weight	
				lbs / 3Mft^2	grams / M^2
Polyester Film Alcohol Resistant Primer LD Polyethylene Totals	ASTM D 3776-85	.48 mils .01 mils 2.00 mils **2.49 mils**	12.0 µ 0.25 µ 50.0 µ **62.25 µ**	10.4 lbs. 0.2 lbs. 28.8 lbs. **39.4 lbs.**	17.0 gms 0.3 gms 46.9 gms **64.2 gms**
CHARACTERISTICS	**Test Method**	**Units**		**Typical**	
MVTR*	ASTM F 372	g/100 si /24 hr.	—	0.42	—
OTR*	ASTM D 3985	cc/100 si /24 hr.	—	9.65	—
Nominal Yield Coated	Tolas	sq. in./lb. M^2/kg	—	10964 15.5	—
Seal Conditions			**Min.**	**Typical**	**Max.**
Temperature		degrees F	210	230	300
		degrees C	99	110	149
Dwell		seconds	.5	1.5	3.0
Pressure		psi	40	50	90

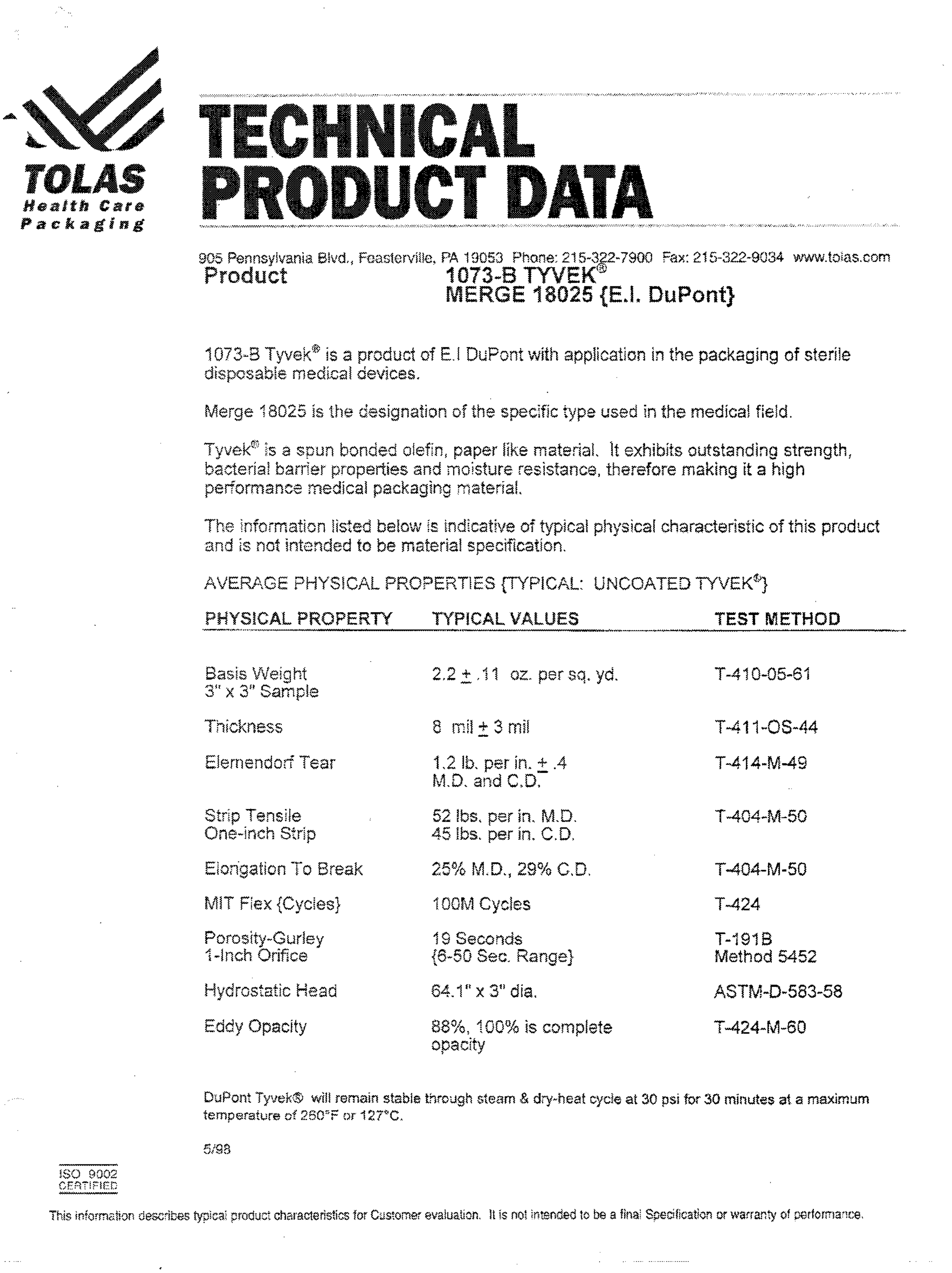
Optimum sealing conditions are highly dependent upon the materials being sealed, the equipment, and production rates. Our recommendation is to begin testing at 240–270°F (115–132°C), 1.5 seconds, 50 psi. Structure weight/thickness may vary by ± 10%.

* Calculated

ISO 9002
CERTIFIED

This information describes typical product characteristics for Customer evaluation. It is not intended to be a final Specification or warranty of performance.

Figure B.1 Material Specification



TOLAS
Health Care
Packaging

TECHNICAL PRODUCT DATA

905 Pennsylvania Blvd., Feasterville, PA 19053 Phone: 215-322-7900 Fax: 215-322-9034 www.tolas.com

Product **1073-B TYVEK®**
MERGE 18025 {E.I. DuPont}

1073-B Tyvek® is a product of E.I DuPont with application in the packaging of sterile disposable medical devices.

Merge 18025 is the designation of the specific type used in the medical field.

Tyvek® is a spun bonded olefin, paper like material. It exhibits outstanding strength, bacterial barrier properties and moisture resistance, therefore making it a high performance medical packaging material.

The information listed below is indicative of typical physical characteristic of this product and is not intended to be material specification.

AVERAGE PHYSICAL PROPERTIES {TYPICAL: UNCOATED TYVEK®}

PHYSICAL PROPERTY	TYPICAL VALUES	TEST METHOD
Basis Weight 3" x 3" Sample	2.2 ± .11 oz. per sq. yd.	T-410-05-61
Thickness	8 mil ± 3 mil	T-411-OS-44
Elemendorf Tear	1.2 lb. per in. ± .4 M.D. and C.D.	T-414-M-49
Strip Tensile One-inch Strip	52 lbs. per in. M.D. 45 lbs. per in. C.D.	T-404-M-50
Elongation To Break	25% M.D., 29% C.D.	T-404-M-50
MIT Flex {Cycles}	100M Cycles	T-424
Porosity-Gurley 1-Inch Orifice	19 Seconds {6-50 Sec. Range}	T-191B Method 5452
Hydrostatic Head	64.1" x 3" dia.	ASTM-D-583-58
Eddy Opacity	88%, 100% is complete opacity	T-424-M-60

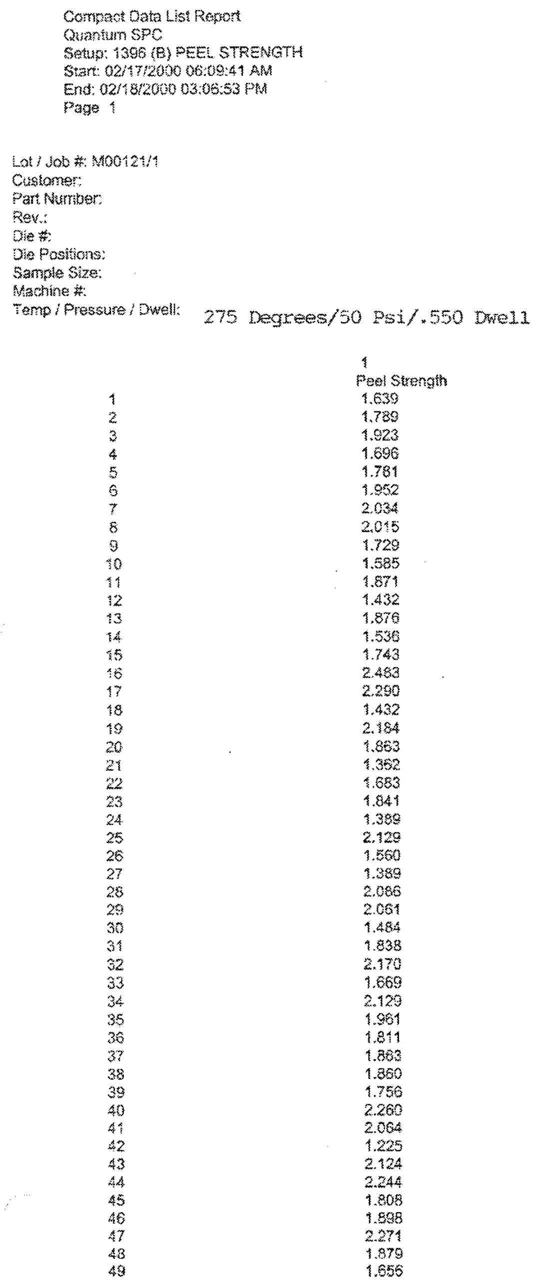
DuPont Tyvek® will remain stable through steam & dry-heat cycle at 30 psi for 30 minutes at a maximum temperature of 260°F or 127°C.

5/98

ISO 9002
CERTIFIED

This information describes typical product characteristics for Customer evaluation. It is not intended to be a final Specification or warranty of performance.

Figure B.2 Material Specification



Compact Data List Report
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End: 02/18/2000 03:06:53 PM
Page 1

Lot / Job #: M00121/1
Customer:
Part Number:
Rev.:
Die #:
Die Positions:
Sample Size:
Machine #:
Temp / Pressure / Dwell: 275 Degrees/50 Psi/.550 Dwell

	1 Peel Strength
1	1.639
2	1.789
3	1.923
4	1.696
5	1.781
6	1.952
7	2.034
8	2.015
9	1.729
10	1.585
11	1.871
12	1.432
13	1.876
14	1.536
15	1.743
16	2.483
17	2.290
18	1.432
19	2.184
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22	1.683
23	1.841
24	1.389
25	2.129
26	1.560
27	1.389
28	2.086
29	2.061
30	1.484
31	1.838
32	2.170
33	1.669
34	2.129
35	1.961
36	1.811
37	1.863
38	1.860
39	1.756
40	2.260
41	2.064
42	1.225
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45	1.808
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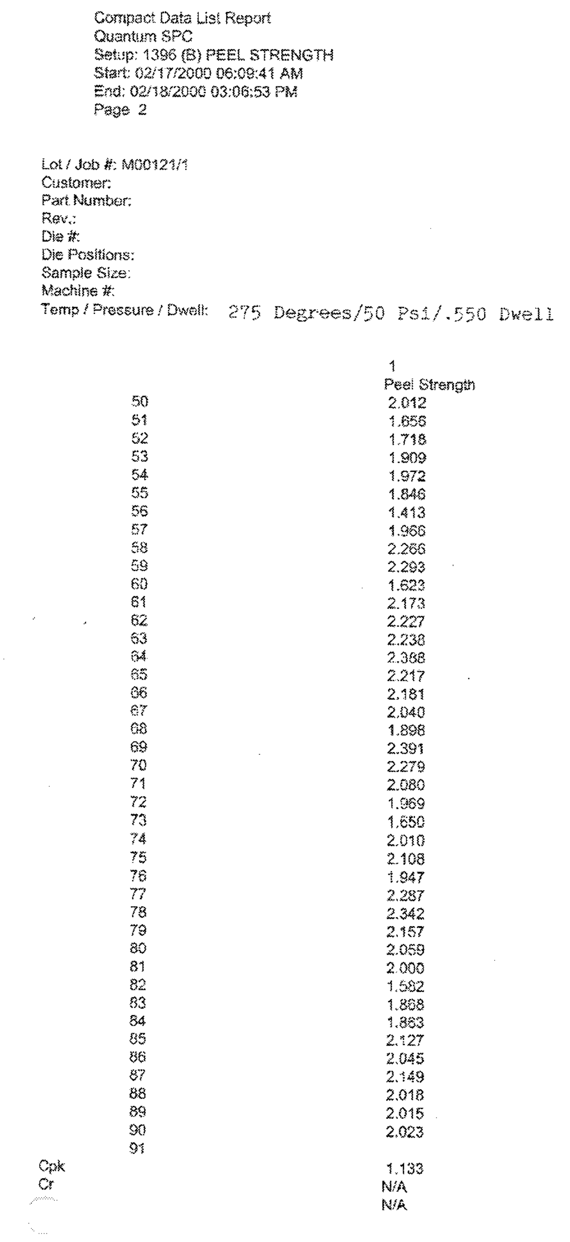
Figure B.3 Material Specification



Compact Data List Report
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End: 02/18/2000 03:06:53 PM
Page 2

Lot / Job #: M00121/1
Customer:
Part Number:
Rev.:
Die #:
Die Positions:
Sample Size:
Machine #:
Temp / Pressure / Dwell: 275 Degrees/50 Psi/.550 Dwell

	1 Peel Strength
50	2.012
51	1.656
52	1.718
53	1.909
54	1.972
55	1.846
56	1.413
57	1.966
58	2.266
59	2.293
60	1.623
61	2.173
62	2.227
63	2.238
64	2.368
65	2.217
66	2.181
67	2.040
68	1.898
69	2.391
70	2.279
71	2.080
72	1.969
73	1.650
74	2.010
75	2.108
76	1.947
77	2.287
78	2.342
79	2.157
80	2.059
81	2.000
82	1.582
83	1.868
84	1.863
85	2.127
86	2.045
87	2.149
88	2.018
89	2.015
90	2.023
91	
Cpk	1.133
Cr	N/A
	N/A

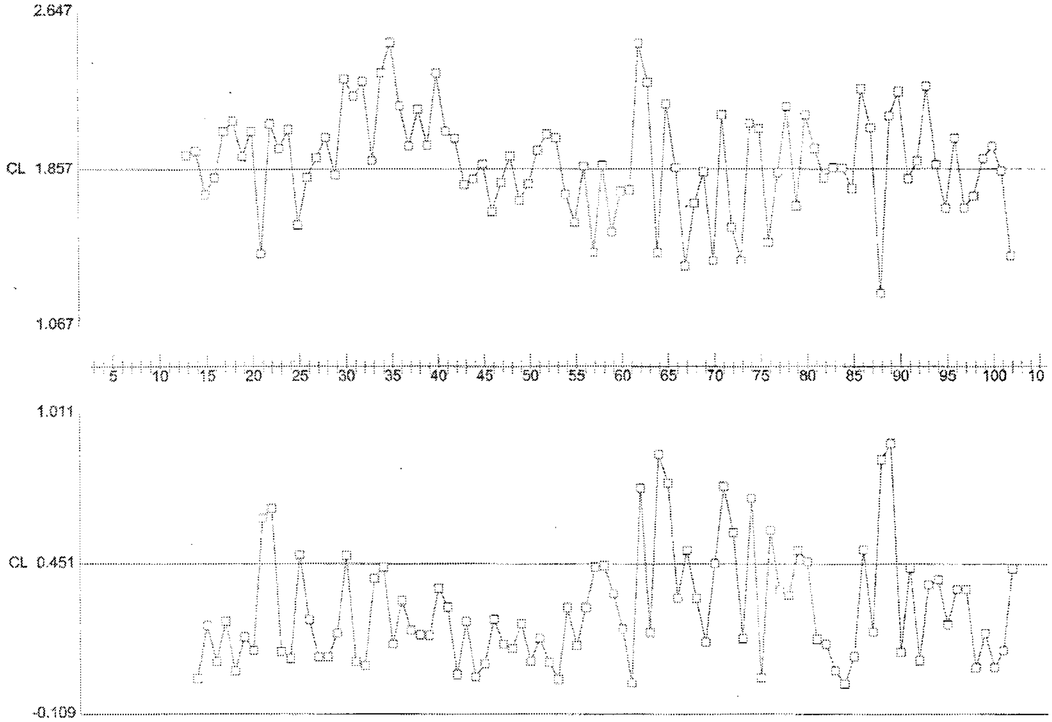
Figure B.4 Material Specification

02/17/2000 11:29:11 PM

Individual & Moving Range

Product: POUCH 0501A / 1073B
Setup: 1396 (B) PEEL STRENGTH
Characteristic: Peel Strength

Tolas Health Care Packaging
Data Analysis Report



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To: 02/17/2000 11:28:46 PM

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R-Bar: 0.2829

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X-Bar CL: 1.8570
X-Bar LCL: NULL

R UCL: NULL
R CL: 0.451
R LCL: NULL

USL: NULL
Nominal: 2.000
LSL: 1.000

Subgroups: 90

Figure B.5 Material Specification

Apendix C

Peel Test Data

Figures C.1 through C.12 display the peel test raw data for the temperature groups of 45°C, 55°C, and 65°C Ambient and 75% RH (including real-time) collected for this research.

1-Year Ambient data

Page 2 of

WESTPAK, INC.
TENSILE TEST DATA

TEST SAMPLE: Peel Seal 65°C DRY 1 YEAR ~~(40 DAYS)~~ **DATE** 4/15/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☐ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21.1 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21.1 °C 35 %RH for ☐ 24 ☐ ___ Hrs

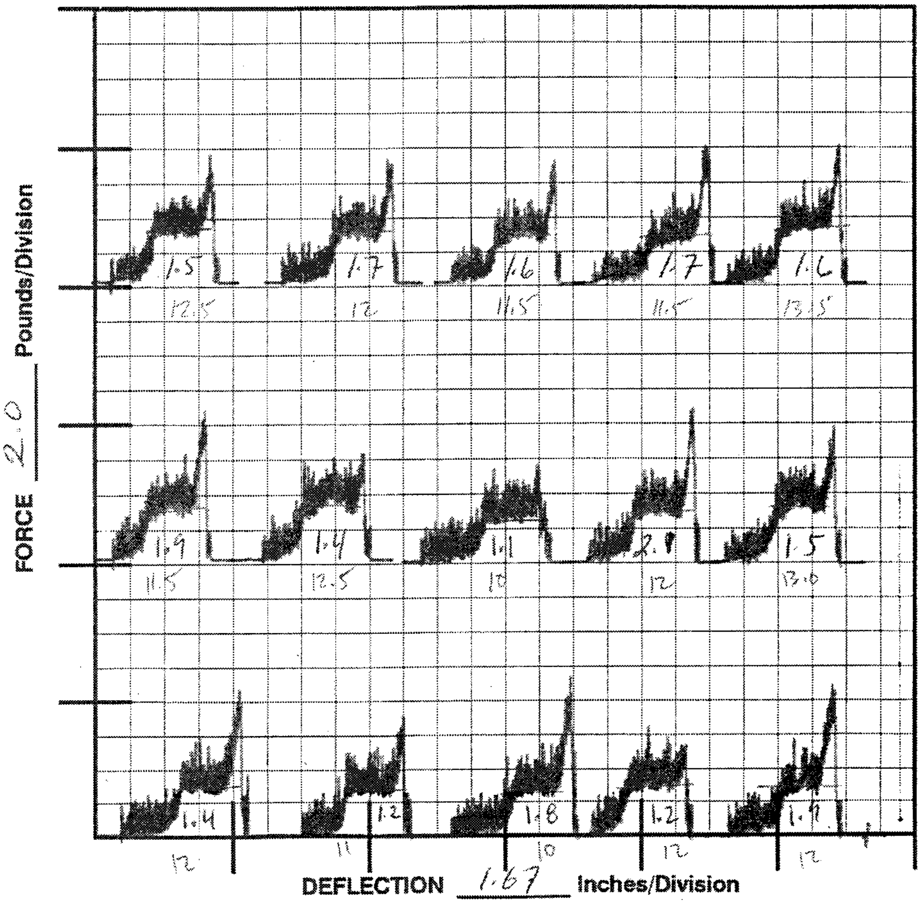

Figure C.1 65°C 1-year

3 of

WESTPAK, INC.
TENSILE TEST DATA

TEST SAMPLE: Peel Seal 65°C Dry 1 YR **DATE** 4/15/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21.1 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21.1 °C 35 %RH for ☒ 24 ☐ ___ Hrs

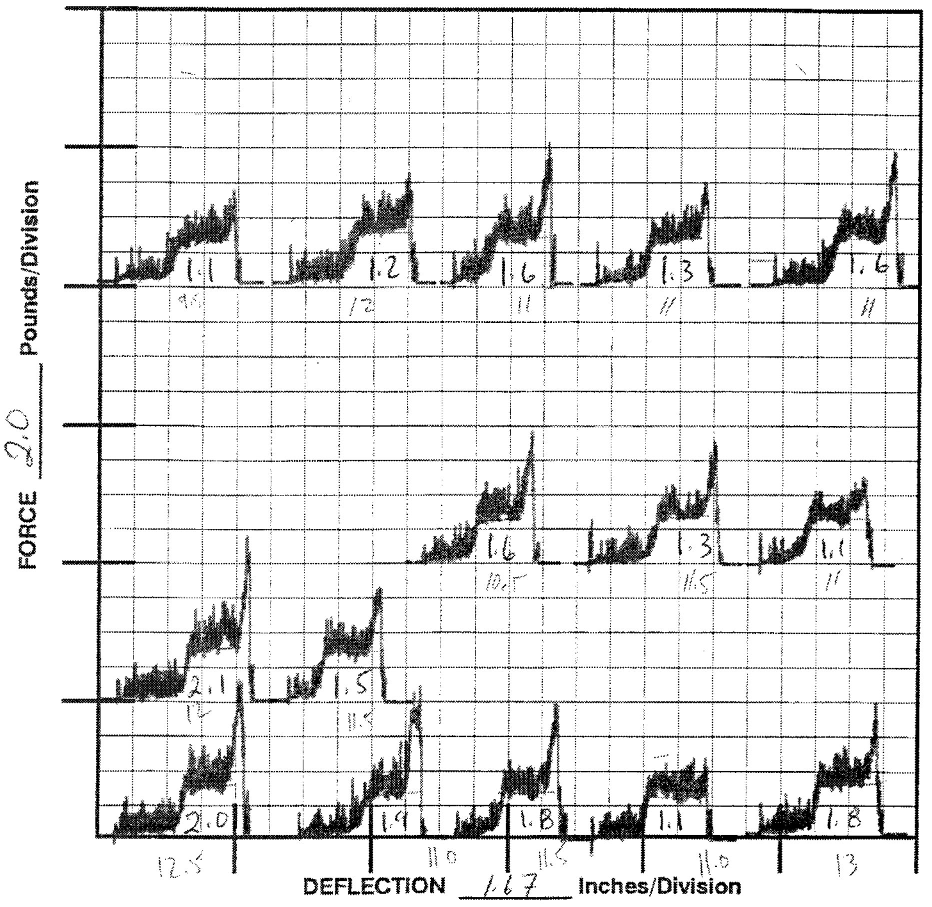

Figure C.2 65°C 1-year cont'

WESTPAK, INC.
TENSILE TEST DATA

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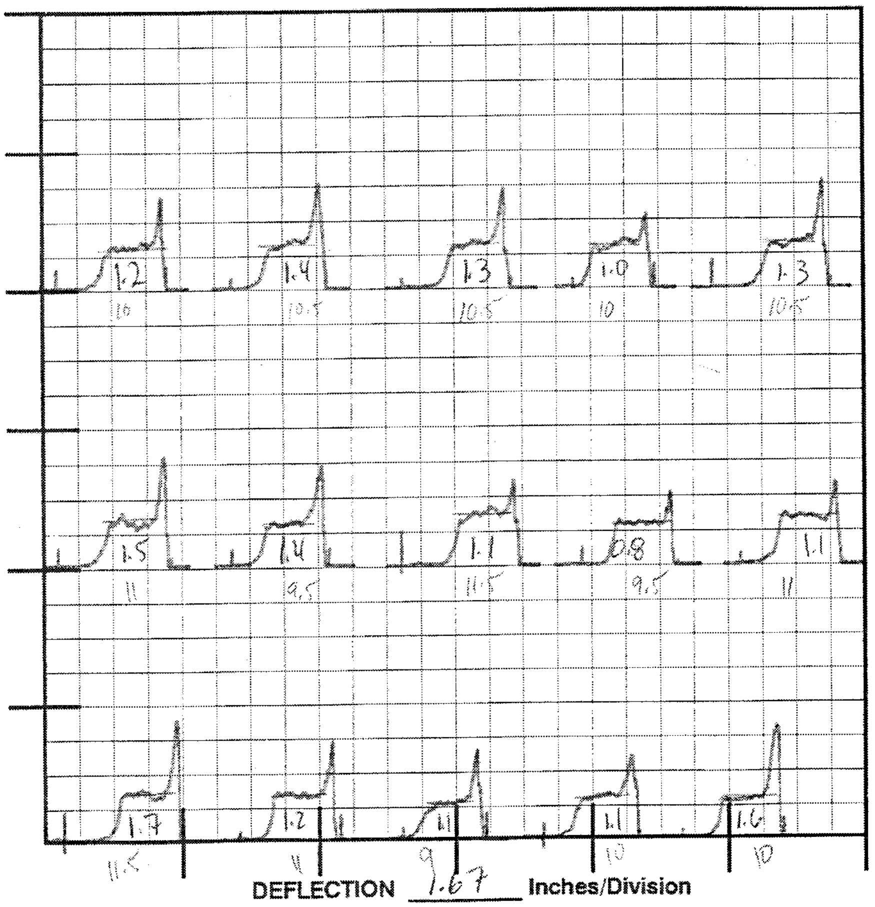
TEST SAMPLE: Peel Seal 55°C Dry 1 YR **DATE** 4/10/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☑ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21 °C 35 %RH for ☒ 24 ☐ ___ Hrs



FORCE 2.0 **Pounds/Division**

DEFLECTION 1.67 **Inches/Division**

Figure C.3 55°C 1-year

WESTPAK, INC.

Pg 12 of

TENSILE TEST DATA

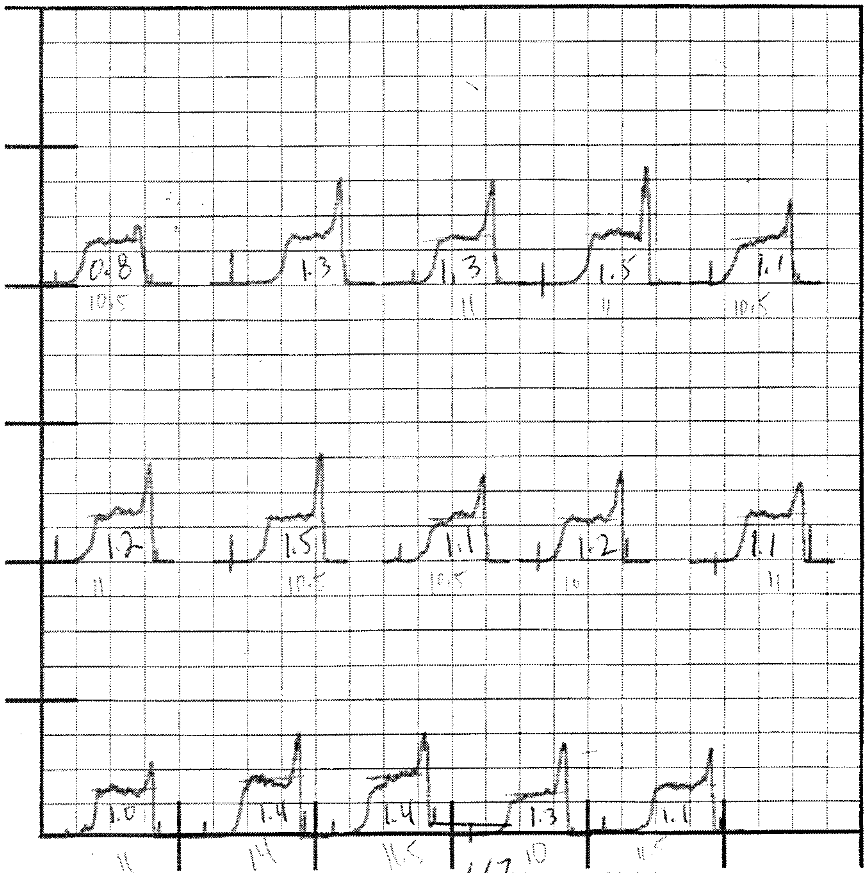
TEST SAMPLE: Peel seal 55°C Dry 1YR Con't **DATE** 4/16/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21 °C 35 %RH for ☒ 24 ☐ ___ Hrs



FORCE 2.0 **Pounds/Division**

DEFLECTION 1.67 **Inches/Division**

Figure C.4 55°C 1-year cont'

WESTPAK, INC.
TENSILE TEST DATA

TEST SAMPLE: Pae Test 45°C DRY 1YR DATE 5/27/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ______

PLATEN SPEED: ☐ .5 in/min. ☐ 10 IN/min

LAB CONDITIONS: ☐ Standard ☐ Ambient ________ Degrees C ________ %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☐ ___°C ___%RH for ☐ 24 ☐ ___ Hrs

1	1.4
2	1.5
3	1.9
4	1.4
5	1.3
6	1.6
7	1.6
8	1.7
9	1.3
10	1.4
11	1.5
12	1.2
13	1.2
14	1.4
15	1.2
16	1.6
17	1.6
18	1.2
19	1.3
20	1.3
21	1.6
22	1.3
23	1.4 -1
24	1.7
25	1.2
26	1.6
27	
28	1.3
29	1.4
30	1.4

Figure C.5 45°C 1-year

1-Year 75% RH data

WESTPAK, INC.
TENSILE TEST DATA

Page 4 of

TEST SAMPLE: Peel Seal ~~60~~ 65°C 75%RH 1YR DATE 4/15/2000
Tested 4/18/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21.1 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21°C 35 %RH for ☒ 24 ☐ ___ Hrs

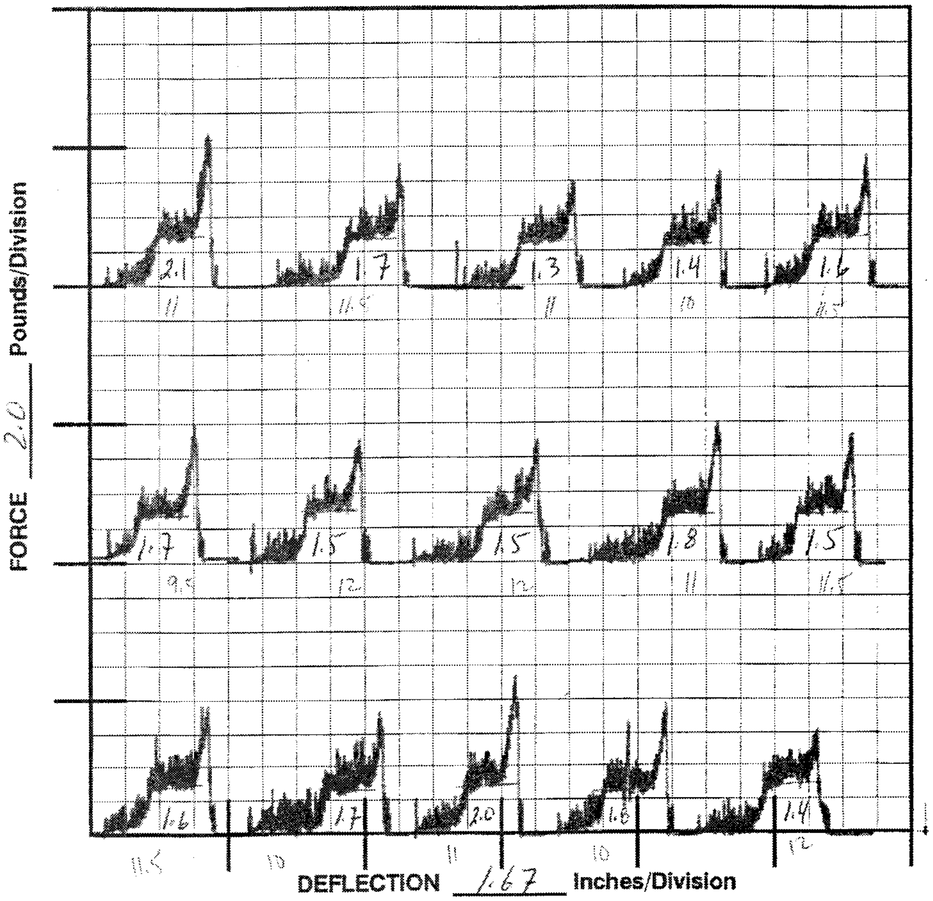


Figure C.6 65°C 1-year 75% RH data

WESTPAK, INC. Pg 5 of

TENSILE TEST DATA

TEST SAMPLE: Peel Seal 65°C 75%RH 1 YR **DATE** 4/15/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21.1 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21.1 °C 35 %RH for ☒ 24 ☐ ___ Hrs

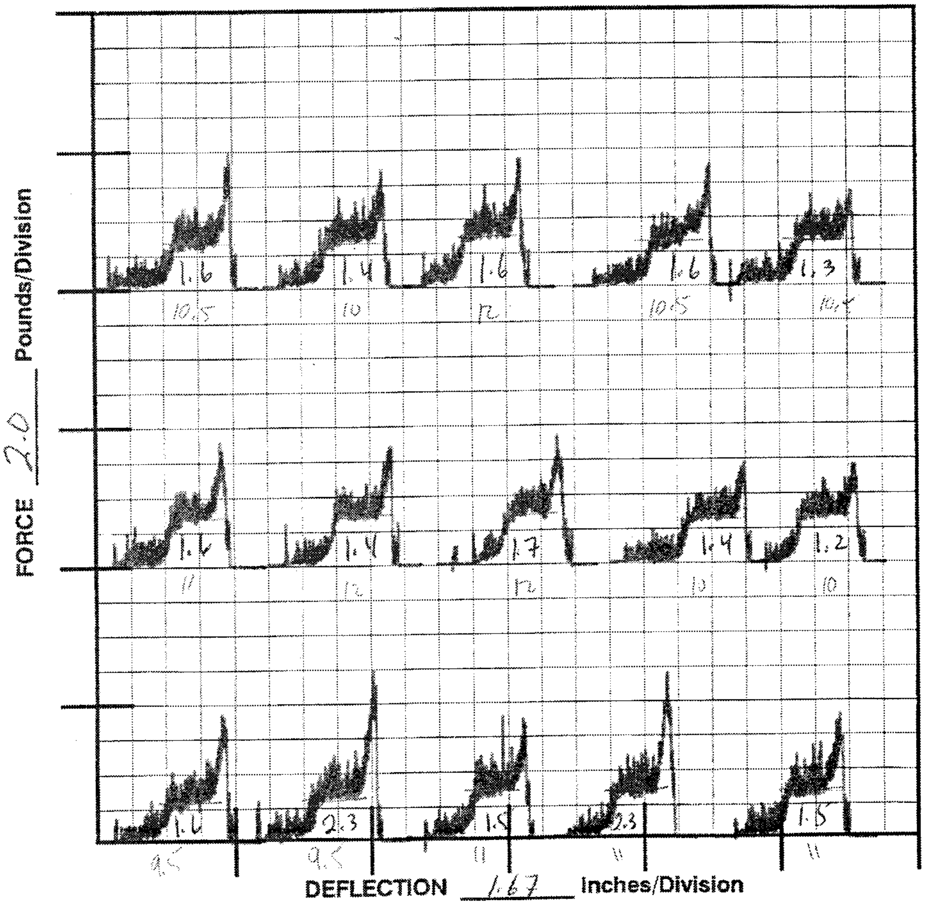


Figure C.7 65°C 1-year 75% RH data cont’

13 9

WESTPAK, INC.
TENSILE TEST DATA

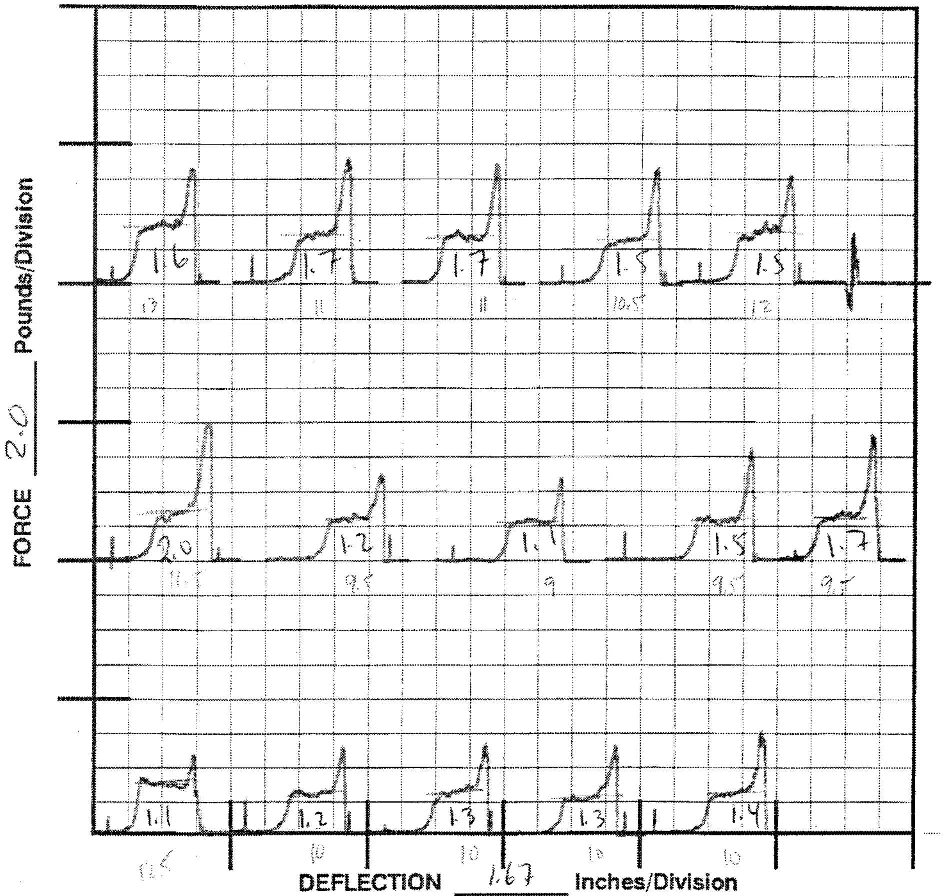
TEST SAMPLE: Peel Seal 55°C 75%RH 1YR **DATE** 4/16/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☒ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☑ 21 °C 35 %RH for ☒ 24 ☐ ___ Hrs



FORCE 2.0 **Pounds/Division**

DEFLECTION 1.67 **Inches/Division**

Figure C.8 55°C 1-year 75% RH data

WESTPAK, INC. 14 of
TENSILE TEST DATA

TEST SAMPLE: Peel Seal 55°C 75% 1YR Con't **DATE** 4/16/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☒ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21 °C 35 %RH for ☐ 24 ☐ ___ Hrs

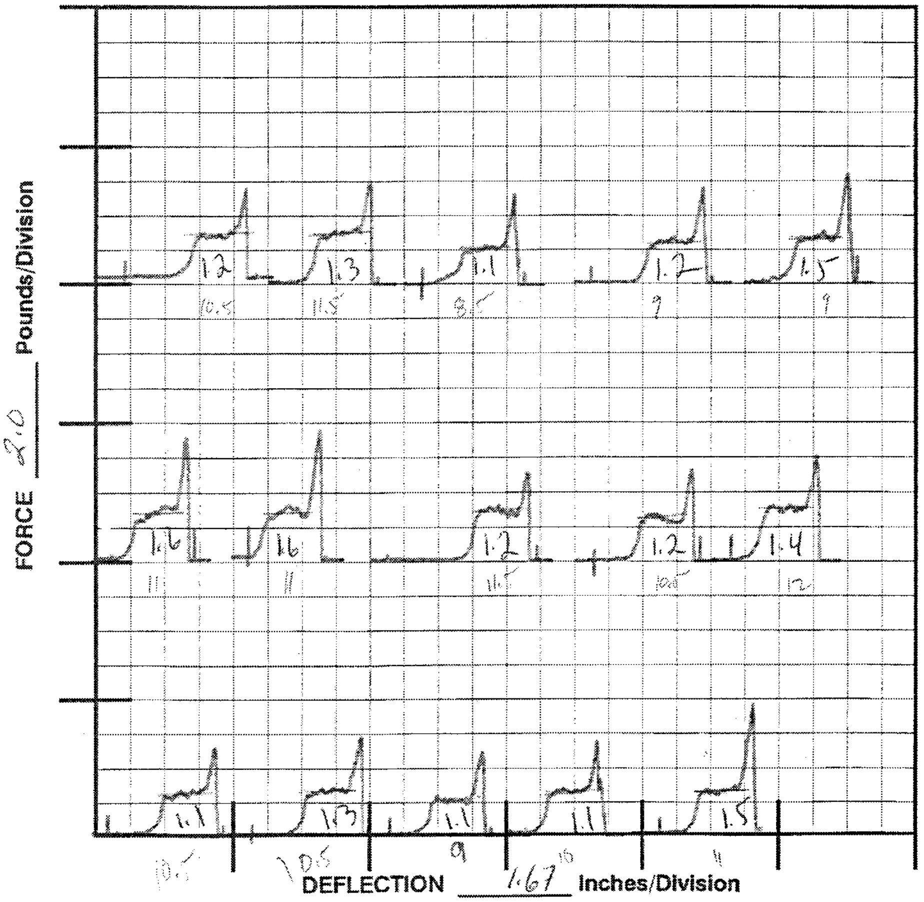


Figure C.9 55°C 1-year 75% RH data cont'

WESTPAK, INC.
TENSILE TEST DATA

TEST SAMPLE: Peel Test 45°C 1YR 75%RH DATE 5/27/2000

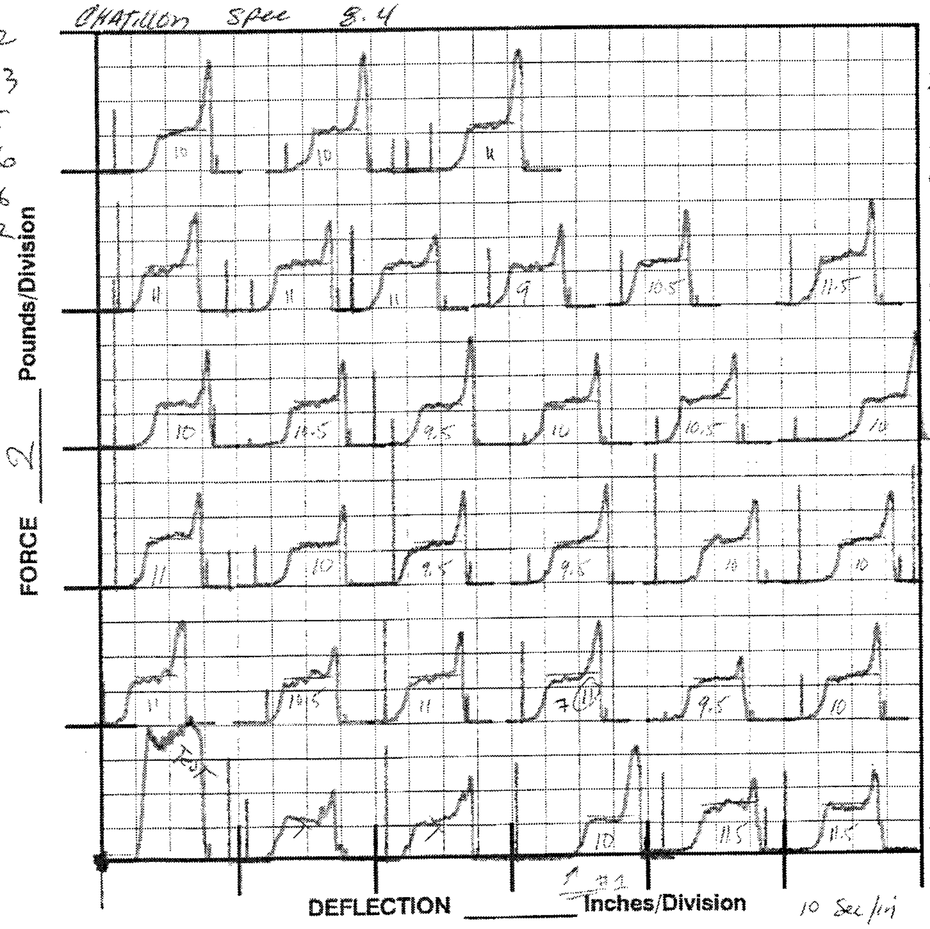
TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ______

PLATEN SPEED: ☐ .5 in/min. ☐ 10 in/min

LAB CONDITIONS: ☐ Standard ☐ Ambient ________ Degrees C ________ %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☐ ___°C ___%RH for ☐ 24 ☐ ___ Hrs

CHATILLON Spec 8.4

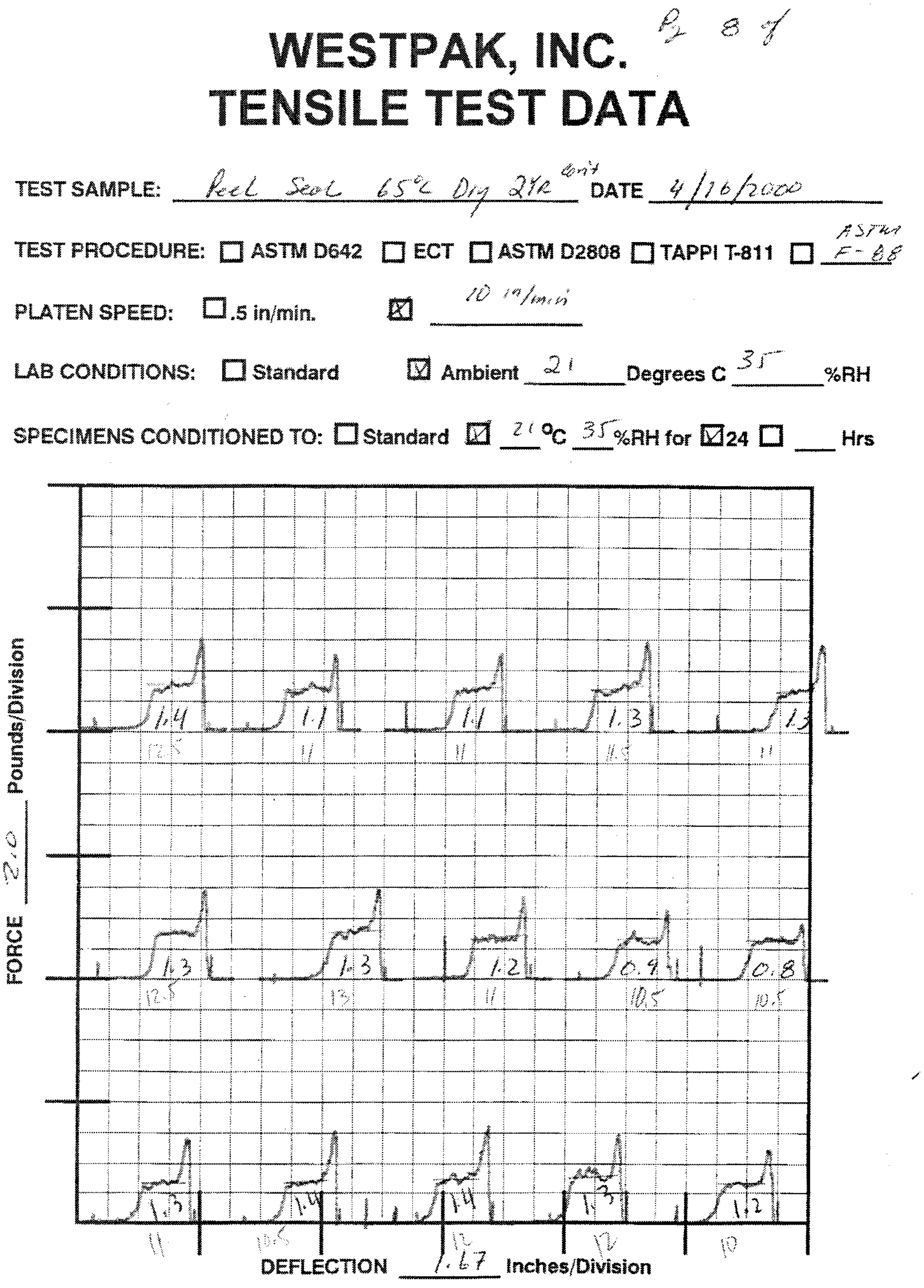


10 Sec/in

1	1.7
	1.2
2	
3	1.2
4	1.5
5	1.1
6	1.3
7	1.4
8	9
9	1.4
10	1.4
11	1.1
12	1.3
13	1.4
14	1.2
15	1.3
16	1.4
17	1.2
18	1.6
19	1.3
20	1.3
21	1.6
22	1.4
23	1.3
24	1.1
25	1.2
26	1.3
27	1.5
28	1.6
29	1.6
30	.7

Figure C.10 45°C 1-year 75% RH data

2-Year Ambient



WESTPAK, INC. Pg 8 of
TENSILE TEST DATA

TEST SAMPLE: Peel Seal 65°C Dry 2YR cont DATE 4/16/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☑ Ambient 21 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☑ 21 °C 35 %RH for ☑ 24 ☐ ___ Hrs

FORCE 2.0 Pounds/Division

DEFLECTION 1.67 Inches/Division

Figure C.11 65°C 2-year Ambient data

WESTPAK, INC. Pg 8 of
TENSILE TEST DATA

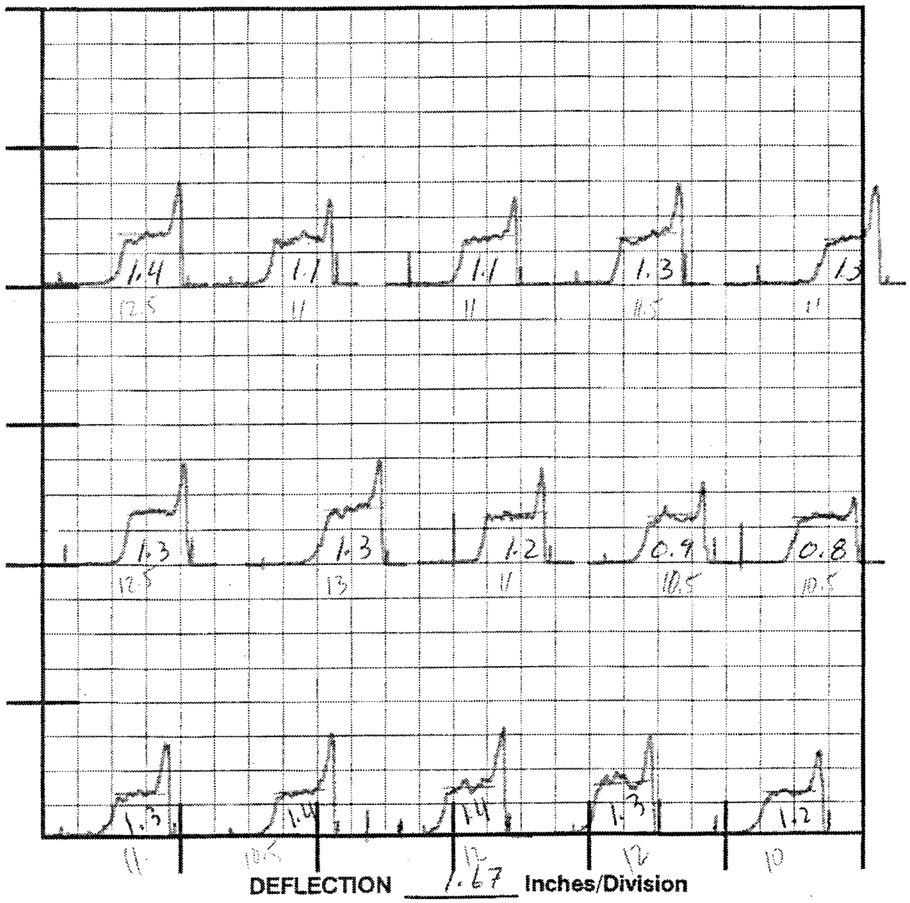
TEST SAMPLE: Peel Seal 65°C Dry 2YR cont DATE 4/16/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21 °C 35 %RH for ☒ 24 ☐ ___ Hrs



FORCE 2.0 Pounds/Division

DEFLECTION 1.67 Inches/Division

Figure C.12 65°C 2-year Ambient data cont’

WESTPAK, INC.
TENSILE TEST DATA

TEST SAMPLE: Peel test 55°C Dry 24R **DATE** 5/27/2000

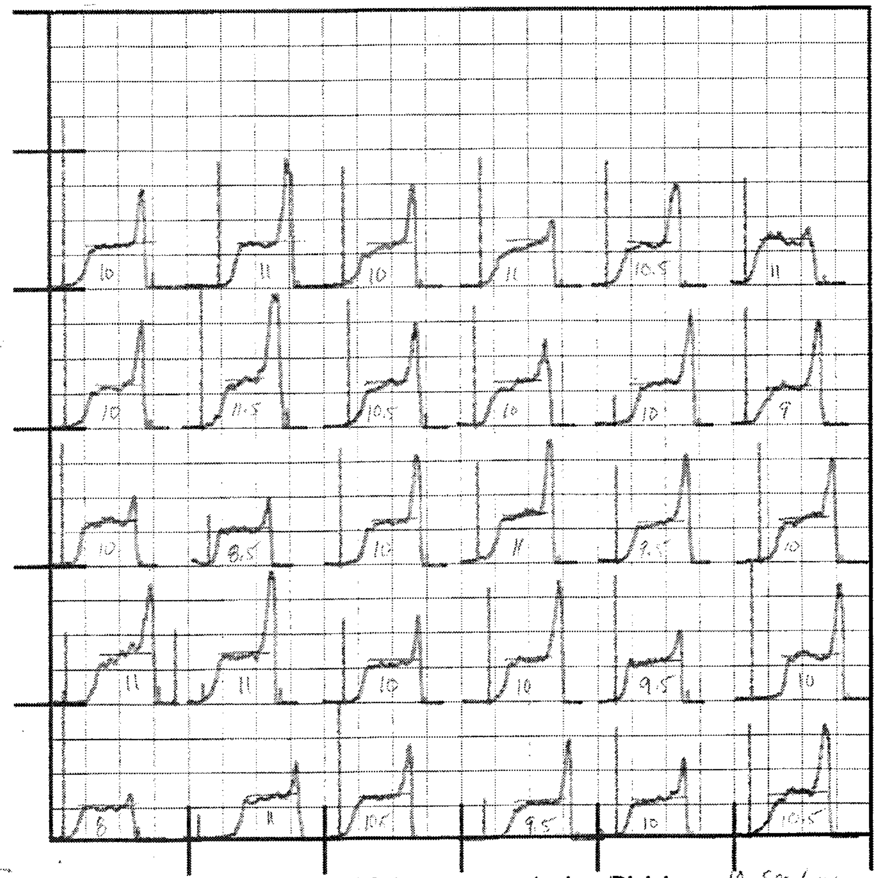
TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ______

PLATEN SPEED: ☐ .5 in/min. ☐ 10 in/sec

LAB CONDITIONS: ☐ Standard ☐ Ambient ________ Degrees C ________ %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☐ ___ °C ___ %RH for ☐ 24 ☐ ___ Hrs

1	.7
2	1.1
3	1.3
4	1.4
5	1.1
6	.6



FORCE 2 **Pounds/Division**

DEFLECTION ________ **Inches/Division** 10 sec/in

7	1.6
8	1.9
9	1.2
10	1.6
11	1.0
12	1.6
13	1.0
14	1.0
15	1.6
16	1.8
17	1.5
18	1.5
19	1.5
20	1.9
21	1.5
22	1.2
23	1.6
24	1.4
25	1.4
26	1.6
27	1.4
28	.9
29	1.5
30	.8

Figure C.13 55°C 2-year Ambient data

Name	Max1_Force	All Mean_Force
Units	lbf	lbf
45C ---%	1.75215	1.52208
1 - 2	2.00390	1.76635
1 - 3	1.72068	1.31608
1 - 4	2.02582	1.81415
1 - 5	1.91287	1.68827
1 - 6	2.01289	1.79167
1 - 7	1.29248	1.10252
1 - 8	1.60885	1.43718
1 - 9	1.80722	1.55687
1 - 10	2.01514	1.71173
1 - 11	2.18260	1.96386
1 - 12	2.34107	1.97128
1 - 13	2.26352	1.99376
1 - 14	1.33125	1.18589
1 - 15	1.93872	1.52772
1 - 16	1.74260	1.55939
1 - 17	1.58918	1.40135
1 - 18	1.73922	1.56003
1 - 19	1.80553	1.59869
1 - 20	2.07639	1.95534
1 - 21	1.65774	1.52330
1 - 22	1.76058	1.52161
1 - 23	1.75721	1.60721
1 - 24	1.66111	1.46853
1 - 25	2.23037	1.63701
1 - 26	1.52456	1.31994
1 - 27	1.45207	1.11940
1 - 28	2.47369	2.20342
1 - 29	2.04380	1.82928
1 - 30	1.62684	1.52145
Mean	1.84500	1.60584
Standard	0.28935	0.26432
Maximum	2.47369	2.20342
Minimum	1.29248	1.10252
Range	1.18121	1.10090

Figure C.14.0 45°C 2-year Ambient data

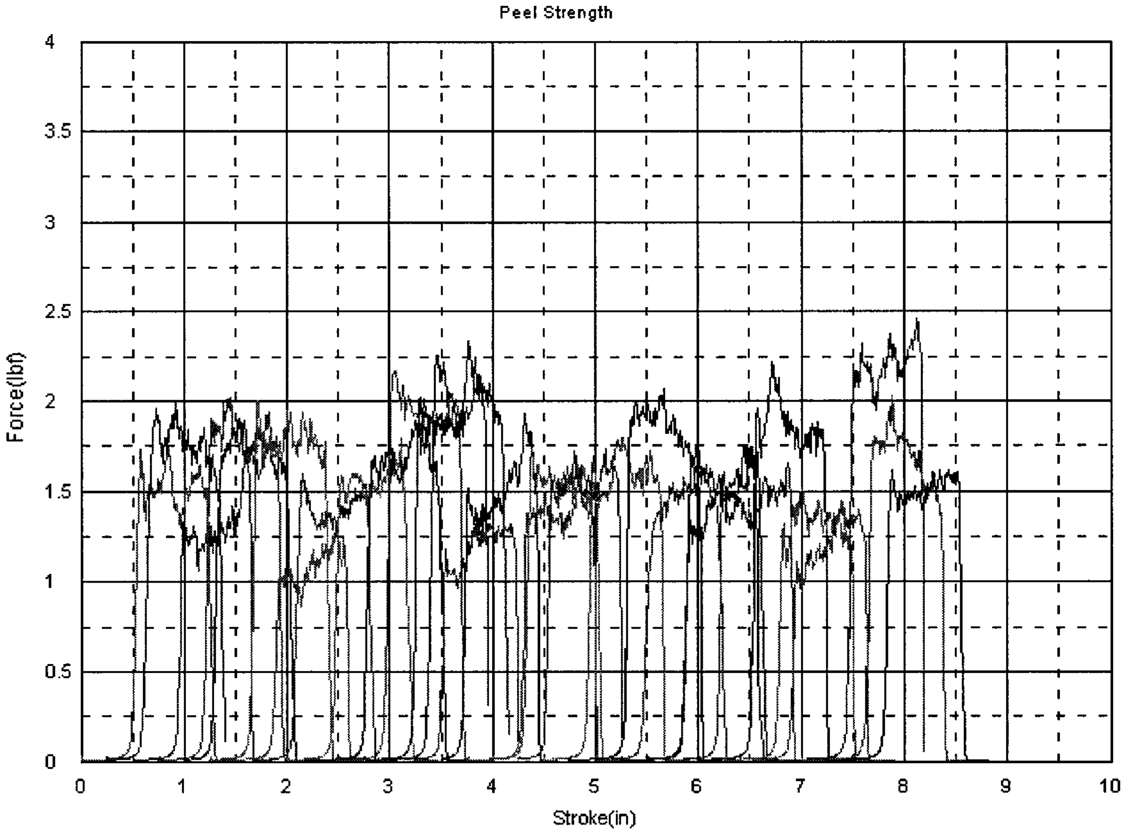


Figure C.14.5 45°C 2-year Ambient data

2-Year 75% RH

Pg 9 of

WESTPAK, INC.
TENSILE TEST DATA

TEST SAMPLE: Peel Seal 65°C 75% 2YR DATE 4/16/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☒ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21 °C 35 %RH for ☒24 ☐ ___ Hrs

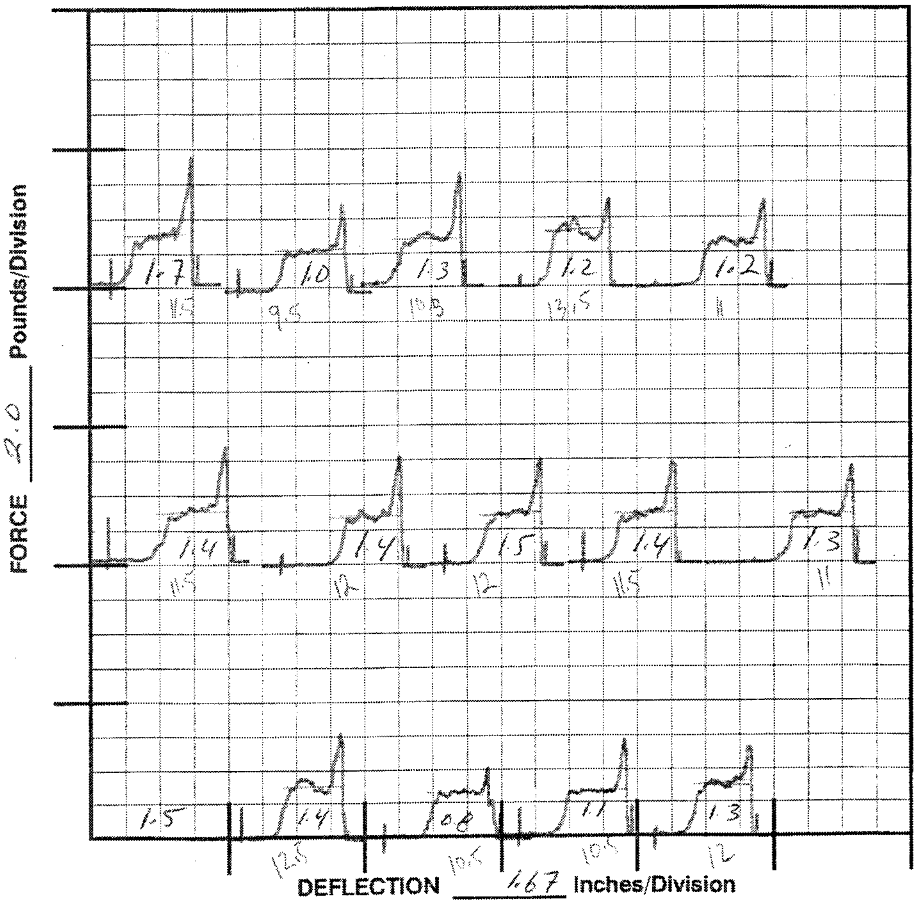
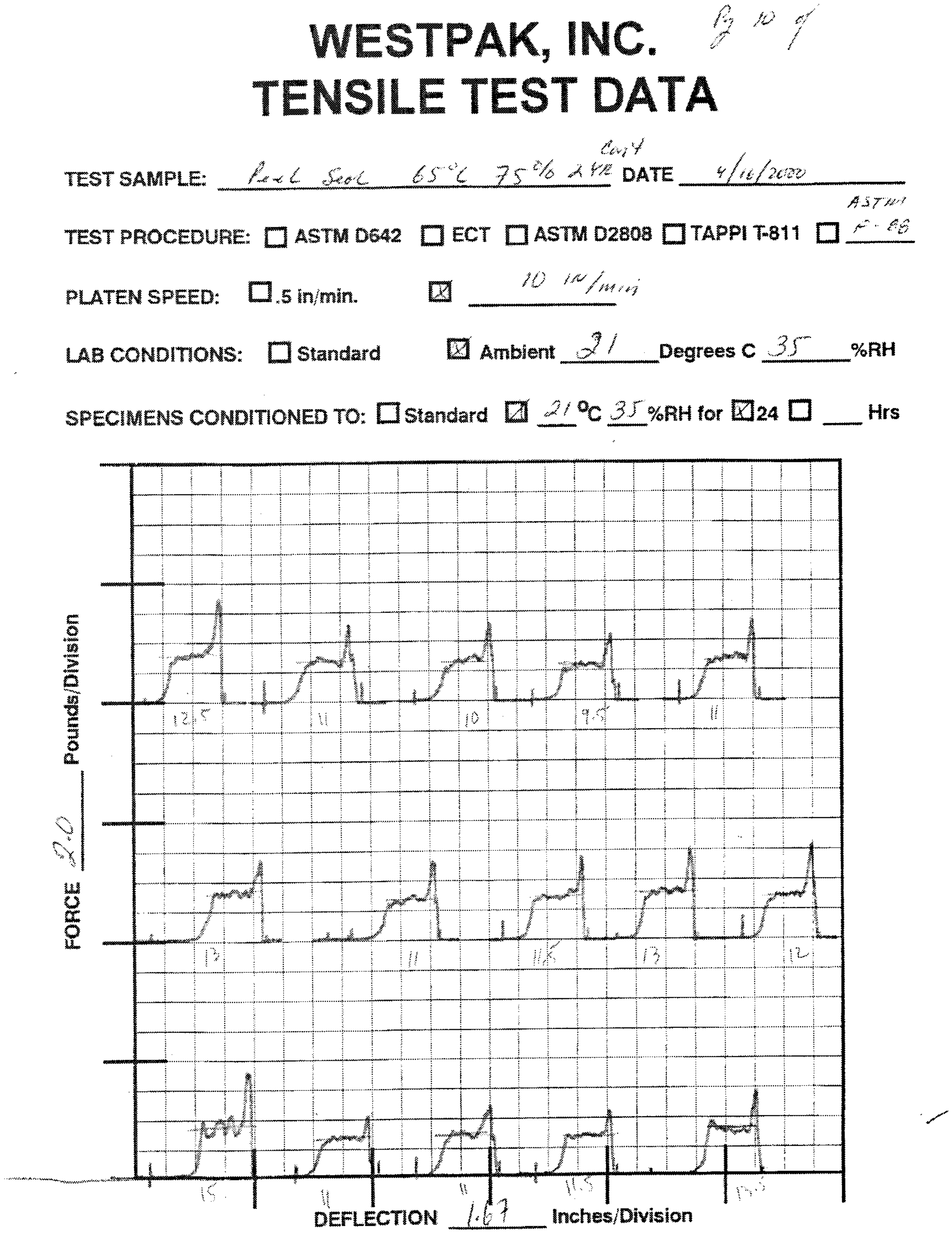


Figure C.15 65°C 2-year 75% RH data



WESTPAK, INC. Pg 10 of
TENSILE TEST DATA

TEST SAMPLE: Peel Seal 65°C 75% 2YR Cont DATE 4/16/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ASTM F-88

PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☒ Ambient 21 Degrees C 35 %RH

SPECIMENS CONDITIONED TO: ☐ Standard ☒ 21 °C 35 %RH for ☒ 24 ☐ ___ Hrs

Figure C.16 65°C 2-year 75% RH data cont'

WESTPAK, INC.
TENSILE TEST DATA

Peel Test
TEST SAMPLE: 55°C 75% 2 YR DATE 5/27/2000

TEST PROCEDURE: ☐ ASTM D642 ☐ ECT ☐ ASTM D2808 ☐ TAPPI T-811 ☐ ______

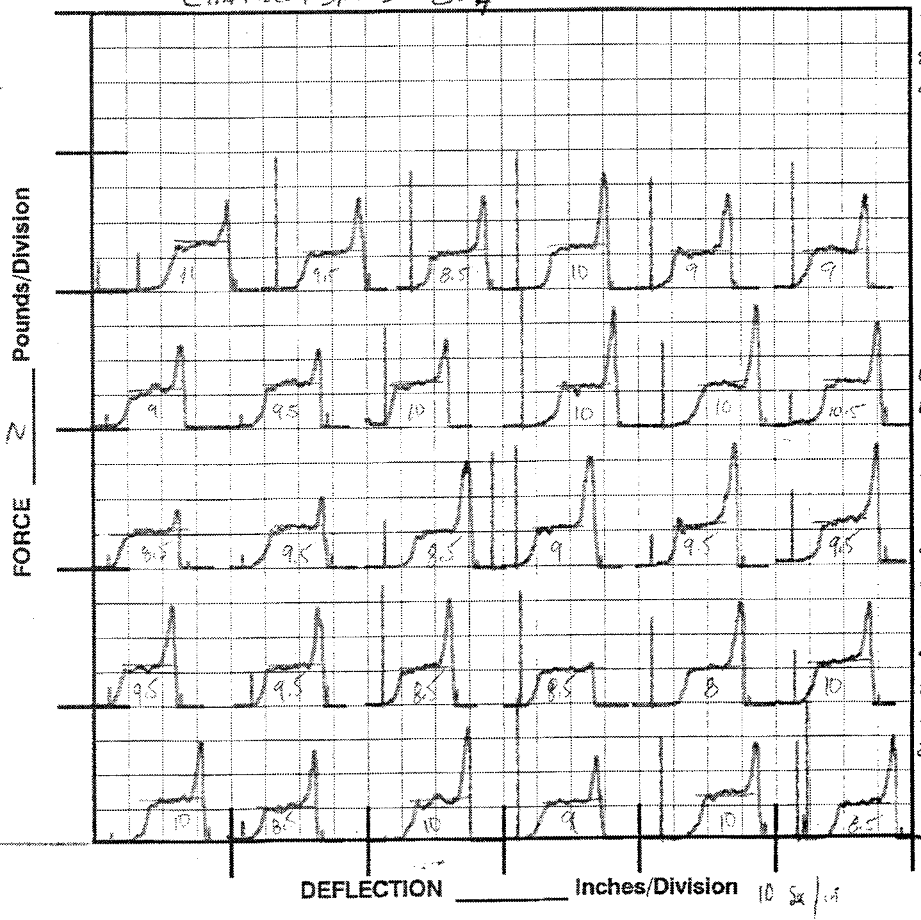
PLATEN SPEED: ☐ .5 in/min. ☒ 10 in/min

LAB CONDITIONS: ☐ Standard ☐ Ambient ________Degrees C ________%RH

SPECIMENS CONDITIONED TO: ☐ Standard ☐ ___°C ___%RH for ☐ 24 ☐ ___ Hrs

Chatillon speed 8.4

1	1.5
2	1.3
3	1.6
4	1.2
5	1.3
6	1.5
7	1.4
8	1.3
9	1.6
10	6
11	1.5
12	1.5
13	7.
14	1.0
15	1.5
16	1.4
17	1.7
18	1.6
19	1.2
20	1.1.
21	1.2
22	1.7
23	1.7
24	1.5
25	1.3
26	1.3
27	1.3
28	1.6
29	1.3
30	1.3



10 Sec/in

Figure C.17 55°C 2-year 75% RH data

Name	Max1_Force	All Mean_Force
Units	lbf	lbf
45C 75% RH -1	1.64650	1.44272
1 - 2	2.24722	1.92177
1 - 3	1.98536	1.79977
1 - 4	2.02638	1.89275
1 - 5	1.25707	1.11854
1 - 6	1.81228	1.59301
1 - 7	1.98367	1.75889
1 - 8	1.88702	1.66845
1 - 9	1.74765	1.52053
1 - 10	2.05110	1.76535
1 - 11	2.14383	1.90078
1 - 12	1.55322	1.21222
1 - 13	2.27869	1.87103
1 - 14	1.79879	1.58469
1 - 15	1.90500	1.44668
1 - 16	1.53299	1.36647
1 - 17	2.08932	1.73538
1 - 18	1.74597	1.50906
1 - 19	2.35793	1.92401
1 - 20	1.46724	1.30898
1 - 21	1.33856	1.14693
1 - 22	1.60042	1.41712
1 - 23	1.70382	1.56592
1 - 24	2.70015	2.56856
1 - 25	2.01458	1.82520
1 - 26	1.94096	1.59506
1 - 27	2.34556	1.85694
1 - 28	2.20733	1.93079
1 - 29	1.42622	1.11181
1 - 30	2.08482	1.80687
Mean	1.89599	1.63888
Standard	0.33511	0.31062
Maximum	2.70015	2.56856
Minimum	1.25707	1.11181
Range	1.44308	1.45675

Figure C.19.0 45°C 2-year 75% RH data

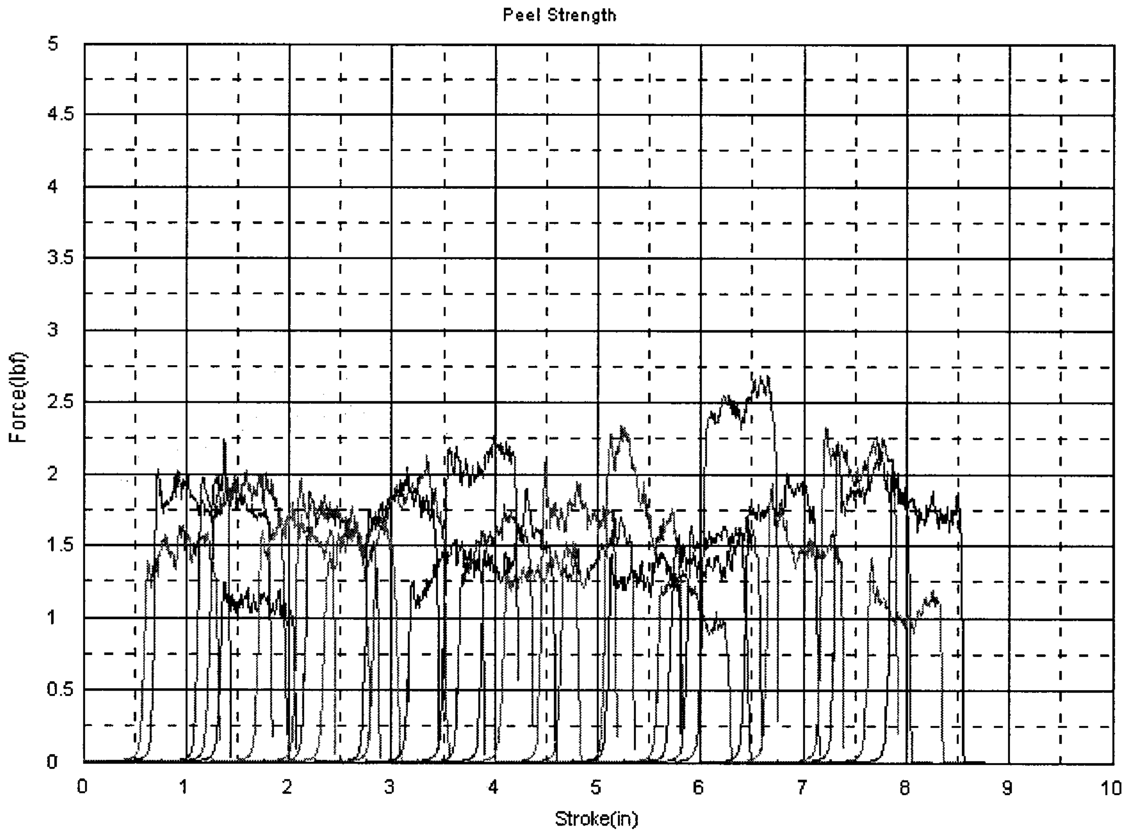


Figure C.19.5 45°C 2-year 75% RH data

3.5 Year Real-Time Data

Name	Max1_Force	All Mean_Force
Units	lbf	lbf
RT 3 yr-1	1.84543	1.68956
1 - 2	2.20564	1.96886
1 - 3	1.99491	1.55132
1 - 4	2.13540	1.98828
1 - 5	2.24273	1.73671
1 - 6	2.31185	1.99102
1 - 7	1.99660	1.70697
1 - 8	2.19103	1.82640
1 - 9	2.46975	2.04218
1 - 10	1.71225	1.47970
1 - 11	2.45852	2.04905
1 - 12	1.48916	1.29067
1 - 13	1.75327	1.57795
1 - 14	1.76620	1.61443
1 - 15	2.40513	2.05180
1 - 16	1.65887	1.46911
1 - 17	2.21519	1.96583
1 - 18	1.74541	1.53805
1 - 19	1.68528	1.47591
1 - 20	1.24246	1.00087
1 - 21	1.81621	1.59553
1 - 22	2.30061	1.98769
1 - 23	2.72375	2.52876
1 - 24	1.74653	1.59541
1 - 25	1.92467	1.55743
1 - 26	2.14045	1.58947
1 - 27	2.05223	1.89619
1 - 28	1.62346	1.41517
1 - 29	2.00109	1.86363
1 - 30	1.68921	1.53999
Mean	1.98478	1.71946
Standard	0.33237	0.29679
Maximum	2.72375	2.52876
Minimum	1.24246	1.00087
Range	1.48129	1.52789

Figure C.20.0 3.5-year Real-Time data

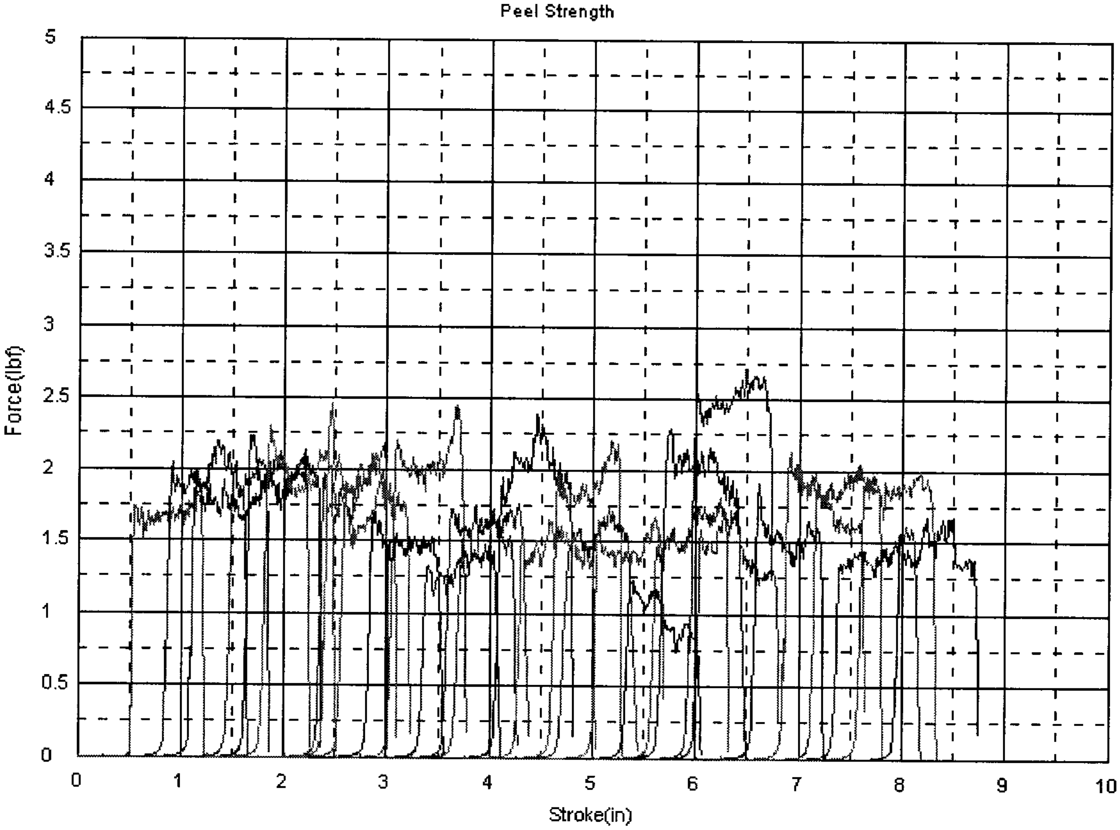


Figure C.20.5 3.5-year Real-Time data