2005

Effect of temperature on removal torque of discontinuous-thread plastic closures

Michael Borchers
San Jose State University

Recommended Citation

Borchers, Michael, "Effect of temperature on removal torque of discontinuous-thread plastic closures" (2005). *Master's Theses*. 2743.
DOI: https://doi.org/10.31979/etd.xj6e-u2na
https://scholarworks.sjsu.edu/etd_theses/2743

EFFECT OF TEMPERATURE ON REMOVAL TORQUE OF DISCONTINUOUS-THREAD PLASTIC CLOSURES

A Thesis
Presented to
The Faculty of the Department of Nutrition and Food Science
San Jose State University

In Partial Fulfillment
of the Requirements for the Degree
Master of Science

by
Michael Borchers
August 2005

UMI Number: 1429406

ProQuest Information and Learning Company
300 North Zeeb Road
P.O. Box 1346
Ann Arbor, MI 48106-1346

APPROVED FOR THE DEPARTMENT

OF NUTRITION AND FOOD SCIENCE

Lucy McProud

Dr. Lucy McProud

Dr. Albert Elboudwarej

Herbert Schueneman

APPROVED FOR THE UNIVERSITY

Rhea I. Williamson

ABSTRACT

EFFECT OF TEMPERATURE ON REMOVAL TORQUE OF DISCONTINUOUS-THREAD PLASTIC CLOSURES

Michael Borchers

Department of Nutrition and Food Science, San Jose State University

This thesis investigates the effect of high and low temperature extremes on the removal torque characteristics of a linerless plastic closure. An initial torque of 12 in-lbs. was applied to polypropylene (PP) and cross-linked high-density polyethylene (HDPE) closures of the same design. After exposure to temperatures ranging from 60°C (140°F) to -18°C (0°F), the closures were removed from the bottles while removal torque was recorded, and percent torque reduction was calculated.

A significant ($p < .01$) torque reduction was observed in the control group, varying from 44.6% to 57.5%. Exposure to high temperature had a noticeable effect on removal torque; the highest torque reduction was 79.4%, found on the PP closures exposed to 60°C. The lowest torque reduction was 35.4%, found on the cross-linked HDPE closure exposed to -18°C. The data indicate that removal torque is greatly affected by exposure to different temperatures, and that closure material is also an important factor.

Acknowledgments

I would like to thank my major professor, Dr. Albert Elboudwarej, for his guidance during my thesis work. I would also like to thank Dr. Lucy McProud and Mr. Herbert Schueneman for their help throughout the course of my study. Your expertise and assistance with my many questions and concerns helped me immensely during this project.

I would also like to thank the staff of Westpak, Inc. of San Jose, California for providing the use of their testing facility, equipment, and on occasion, an extra set of hands.

Finally, I would like to thank Megan, my family, and many friends who have offered their support and encouragement during this challenging and rewarding experience.

Preface

The following is a publication style thesis. The first and third chapters are written according to guidelines outlined in the *Publication Manual of the American Psychological Association*, 5th edition, 2001. Chapter two is written in journal format for submission to *Packaging Technology and Science*.

Table of Contents

List of Tables

List of Figures

List of Acronyms, Abbreviations, and Variables

%R	Percent Reduction in Torque
ASTM	American Society for Testing and Materials
°C	Celsius Degrees
°F	Fahrenheit Degrees
HDPE	High Density Polyethylene
in.-lbs	Inch-Pounds Torque
ml	Milliliters
PET	Polyethylene Terephthalate
PP	Polypropylene
RH	Relative Humidity
T	Temperature
ΔT	Temperature Change
T_A	Application Torque
T_R	Removal Torque

CHAPTER 1:

Introduction and Literature Review

Introduction

Significance

It is widely accepted that the closure is one of the most critical parts of a package, and that cap tightness, or torque, is the most important factor in the security of the closure (Hanlon, 1992). If a cap requires too little removal torque, it could mean that the seal was not tight enough, and product damage may result from oxygen or water vapor entering the package (Nairn & Norpell, 1997). Most pharmaceutical products are very sensitive to water vapor, so this situation is clearly undesirable. On the other hand, having too high a torque could make it difficult for the consumer to remove the cap (Hanlon, 1992). This is especially true for the elderly, or for those suffering from arthritis.

Plastic closures on plastic containers tend to lose a considerable amount of their application torque simply due to the viscoelastic nature of the materials involved (Soroka, 2002). Elevated temperatures can cause additional torque loss due to expansion of the cap (Hanlon, 1992). Cross-country shipment or weeks of storage in a non-insulated warehouse can expose the package system to a broad range of temperatures. So the question is, how does exposure to different temperature extremes affect removal torque over time? Clearly, this is an important consideration for packaging engineers.

Research Hypotheses

It is generally understood that exposure to high temperature affects many materials, often causing some degree of expansion, and this is certainly the case with many polymeric plastics. Exposure to cold temperature should have a much smaller effect, at least with regards to any change in size of the bottles or caps. Therefore, based on prior experiments (Lockhart, 1992; Greenway, 1993; Lai & Greenway, 1999; Pisuchpen, 2000), and on a general understanding of the plastic materials used in this experiment, several research hypotheses can be formed.

First, it is predicted that samples exposed to a high temperature environment will experience a statistically significant reduction in cap removal torque compared to control samples. Second, it is predicted that samples exposed to a low temperature environment will not experience a significant reduction in removal torque compared to control samples. For samples exposed to a combination of high and low temperatures, two additional hypotheses can be made. First, it is predicted that these samples will experience a statistically significant reduction in removal torque compared to control samples. Second, it is predicted that these samples will not experience a significant reduction in removal torque compared to samples from the group exposed only to high temperature.

Literature Review

All packaged products are vulnerable to many forms of deterioration, such as ingress of water vapor or oxygen, and the package is further challenged by extreme heat and cold, and by stresses imposed by the distribution cycle (Nairn & Norpell, 1997). "The smallest part of a package, and often the most critical, is usually the closure. The security of the whole assembly and the integrity of the contents are dependent upon the cap or tie or whatever is used to complete the package" (Hanlon, 1992). How well the closure keeps the bottle contents secure can depend on a number of things, such as liner material or flatness of sealing surface, but the most important factor is application tightness, or torque (Hanlon, 1992). Application and removal torque are measurements, in inch-pounds force (in.-lbs), of the maximum amount of force tending to cause rotation of a closure over the neck finish of a container, causing the closure to be secured or unsecured from the container (ASTM, 2002). Application torque typically drops off, however, and the removal torque, or the amount of force necessary to loosen and remove the closure, is lower than application torque (Soroka, 2002). This is due to an effect known as creep, which is the tendency of a material to stretch or sag over time when a steady load is applied (Khol, 1999).

Plastic bottles and caps are increasingly popular, especially in the pharmaceutical industry. A recent study by the Freedonia Group, Inc. predicted that plastic bottles would represent one of the fastest growing segments of the pharmaceutical industry, with demand increasing 6.5% to a total of $130 million by 2006 (Van Houten, 2004). Some of this popularity is undoubtedly due to the low cost and good moisture barrier properties of HDPE (Hanlon, 1992, Soroka, 2002). Polypropylene is also inexpensive, and boasts good physical properties such as stiffness, tensile strength, and temperature resistance, so that most threaded closures are made from PP (Soroka, 2002). These plastics also have the added benefit of being very resilient, allowing the use of internally molded sealing structures such as projecting ridges or vanes to create a linerless closure (Soroka, 2002). Thus, studies involving linerless closures are quite relevant to the packaging industry.

Torque testing is often performed following the procedure outlined in the American Society for Testing and Materials (ASTM) standard D 2063-91, Standard Test Method for Measurement of Torque Retention for Packages with Continuous Thread Closures (ASTM, 2002). In 1992, Dr. Hugh Lockhart studied the effects of different temperatures on the removal torque of a 28mm metal closure on a plastic bottle (Lockhart, 1992). For samples with an initial torque of 13.0 in.-lbs, he found an average torque loss of 3.8% for samples at a low temperature (5°C), and 13.8% for samples at ambient temperature (23°C). The most significant reduction, however, occurred at a high temperature (43°C), where samples lost an average of 75.4% of their application torque (Lockhart, 1992). This is not too surprising, because plastics are more susceptible to creep than metals, and will exhibit some deformation even at room temperature, but will stretch even more at elevated temperatures (Khol, 1999).

In 1993, Dr. Gerald Greenway studied the effect of time on removal torque using bottles made of polyethylene terephthalate (PET), with 24mm HDPE closures and paper pulp liners (Greenway, 1993). Over the course of ten days at room temperature, he observed torque loss that was logarithmic in nature; torque decreased rapidly at first, then continued to decrease more and more slowly over time (Greenway, 1993). In 1999, Dr. Ching-Sung Lai and Dr. Gerald Greenway studied the effect of time on removal torque using bottles made of PET, with 28mm closures made of PP and a vinyl liner material (Lai & Greenway, 1999). They found that removal torque increased for approximately the first ten days, then slowly decreased. Lai and Greenway concluded that there was a surface friction interaction between the bottle finish and the vinyl liner of the closure, which caused the initial increase in torque. This interaction became weaker after ten days, resulting in the expected eventual decrease in removal torque (Lai & Greenway, 1999).

In 2000, Supachai Pisuchpen wrote a Masters thesis on a model for predicting application torque and removal torque of a continuous thread closure (Pisuchpen, 2000). He examined forces and moments caused by the geometry and construction of several different closures, liner materials, and the bottle finishes. He concluded that sealing force is a better indicator of seal strength than removal torque, but also noted that the predictive model did not account for viscoelastic properties or the function of time. (Pisuchpen, 2000). In 2002, Pisuchpen followed up his earlier work with a Doctoral dissertation on measuring and modeling the effect of time and temperature on removal torque and sealing force of a continuous thread closure (Pisuchpen, 2002). He developed a system to measure sealing forces using a strain gage based transducer, and compared this to the more traditional method of removal torque measurement. He found that while the sealing force method was more complicated to set up, it provided more consistent results than measuring removal torque (Pisuchpen, 2002).

CHAPTER 2:

Journal Article

Author's Title Page

Full Title: EFFECT OF TEMPERATURE ON REMOVAL TORQUE OF DISCONTINUOUS-THREAD PLASTIC CLOSURES

Short Title: Temperature Effects on Torque and Bottle Opening

Authors: Michael Borchers – Graduate Student, San Jose State University
One Washington Square, San Jose, California 95192-0058
Ph: 408-924-3100, Fx: 408-924-3114, xuan@casa.sjsu.edu

Albert Elboudwarej, PhD – Adjunct Professor, San Jose State University, Thesis Advisor

Herbert Schueneman, CPP-MH – Packaging Program Director, San Jose State University

Lucy McProud, PhD, RD – Chair of Nutrition & Food Science Department, San Jose State University

Abstract

This thesis investigates the effect of high and low temperature extremes on the removal torque characteristics of a linerless plastic closure. An initial torque of 12 in-lbs. was applied to polypropylene (PP) and cross-linked high-density polyethylene (HDPE) closures of the same design. After exposure to temperatures ranging from 60°C (140°F) to -18°C (0°F), the closures were removed from the bottles while removal torque was recorded, and percent torque reduction was calculated.

A significant ($p < .01$) torque reduction was observed in the control group, varying from 44.6% to 57.5%. Exposure to high temperature had a noticeable effect on removal torque; the highest torque reduction was 79.4%, found on the PP closures exposed to 60°C. The lowest torque reduction was 35.4%, found on the cross-linked HDPE closure exposed to -18°C. The data indicate that removal torque is greatly affected by exposure to different temperatures, and that closure material is also an important factor.

Keywords: Removal Torque, Temperature, Closure, Discontinuous-Thread

Introduction

All packaged products are vulnerable to deterioration, such as ingress of water vapor, and the package is further challenged by extreme heat and cold, and by stresses imposed by the distribution cycle [1]. "The smallest part of a package, and often the most critical, is usually the closure. The security of the whole assembly and the integrity of the contents are dependent upon the cap or tie or whatever is used to complete the package" [2]. How well the closure keeps the bottle contents secure can depend on a number of things, but the most important factor is application tightness, or torque [2]. Application and removal torque are measurements of the maximum amount of force tending to cause rotation of a closure over the neck finish of a container, causing the closure to be secured or unsecured from the container [3].

Application torque typically drops off, and the removal torque, or the amount of force necessary to loosen and remove the closure, is lower than application torque [4]. Elevated temperatures can cause additional torque loss due to expansion of the cap [2]. After a bottle is sealed, it might be several days or even weeks before it reaches the end user, and the product might experience great variations in temperature during cross-country distribution, or during storage in a non-insulated warehouse. So the question is, how much torque loss will occur due to extreme temperature conditions? This is an important question for the packaging engineer to consider.

Plastic bottles and caps are increasingly popular. A recent study by the Freedonia Group, Inc. predicted that plastic bottles would represent one of the fastest growing segments of the pharmaceutical industry [5]. Some of this popularity is undoubtedly due to the low cost and good moisture barrier properties of HDPE and PP, as well as their good physical properties and temperature resistance [2] [4]. Polypropylene has the added benefit of being very resilient, allowing the use of internally molded sealing structures such as projecting ridges or vanes to create a linerless closure [4]. Thus, studies involving linerless closures are quite relevant to the packaging industry.

Several experiments have already been conducted in this area, but all that could be found in a review of literature involved continuous-thread closures. In 1992, for example, Dr. Hugh Lockhart studied the effects of temperature on the removal torque of a 28mm metal closure on a plastic bottle [6]. For samples with an initial torque of 13.0 in.-lbs, he found the greatest torque reduction at a high temperature (43°C), where samples lost an average of 75.4% of their application torque [6]. In 1999, Dr. Ching-Sung Lai and Dr. Gerald Greenway studied the effect of time on removal torque using bottles made of polyethylene terephthalate (PET), along with 28mm closures made of PP with a vinyl liner material [7]. They found that removal torque increased for the first ten days before eventually decreasing, but concluded that this initial increase was caused by a surface friction interaction between the bottle finish and the vinyl liner of the closure [7].

In 2000, Supachai Pisuchpen wrote a Masters thesis on a model for predicting application and removal torque of a continuous thread closure [8]. By examining forces and moments caused by the geometry and construction of different closures, he concluded that sealing force is a better indicator of seal strength than removal torque, but also noted that the predictive model did not account for viscoelastic properties or the function of time [8]. In 2002, Pisuchpen followed up his earlier work with a Doctoral dissertation on measuring and modeling the effect of time and temperature on removal torque and sealing force of a continuous thread closure [9]. He developed a system to measure sealing forces, and compared this to the more traditional method of removal torque measurement. He found that the sealing force method provided more consistent results than measuring removal torque [9]. For the experiment described in this paper, the more traditional method of torque measurement was used, due to availability of the necessary equipment, and the straightforward, repeatable nature of the procedure.

Materials and Equipment

Test Specimens

The specimens used in this experiment were black 50ml round bottles made of Marlex BN injection blow-molded high-density polyethylene (HDPE), obtained from Sanner of America, with an average weight of 11.1g each. The bottles had a 28mm diameter finish, with a 1/3-turn discontinuous thread. Most plastic bottles have either continuous threads or lug style threads. Continuous threads, as the name implies, are molded continuously around the entire bottle finish, sometimes making several revolutions. Conversely, lug style threads have only a short length, and are well known for their use on jars for mayonnaise and other food products [2]. Examples of both continuous threads and lug style threads are shown in Figure 1. Discontinuous threads have a profile and pitch similar to continuous threads, but are only molded a portion of the way around the bottle. On the samples used in this experiment, each thread covers 120 degrees, or one third of a complete revolution. This hybrid design combines the positive sealing capability of continuous threads with the ease of opening found in lug threads.

Half of the closures used were white injection-molded Huntsman H1200 polypropylene (PP) desiccant caps, as are commonly used in pharmaceutical applications. The closures had a 1/3-turn discontinuous thread to match the bottles, and were designed with a protruding fin that would seal to the inside of the bottle finish without the need for a liner. The remaining closures were also desiccant caps of the same design, but made of cross-linked HDPE. Normally, HDPE resin is composed of linear molecules that can pack together very tightly, giving the plastic many of its desirable physical properties [10]. In cross-linked HDPE, the long polymer chains are joined chemically at multiple tie points, forming a web-like structure that is more resistant to heat [10]. All caps had their desiccant material removed prior to testing. Cross-linked HDPE caps without desiccant had an average weight of 3.36g each, and PP caps without desiccant had an average weight of 3.32g each.

The size of each sample group was large (n = 30) to allow for variations in bottle or cap manufacture. All bottles were obtained in a single batch from the same manufacturer; however, there were still several different molds. This occurs because several bottles are made at once in a multi-cavity mold, each marked with a number indicating its position. In this case, bottles received were marked with numbers ranging from 22 to 33, and each sample group consisted of bottles from several different cavities. An alternate method of assigning bottles to groups was also considered, wherein each group would consist entirely of bottles with the same mold number. This idea was rejected however, as it would not allow for possible minor variations from one mold to another. Having different mold numbers within each sample group took any such variations into account.

Equipment

For measurement of application and removal torques, a calibrated digital torque meter was used. The meter was a Kaps-All model EB-550, obtained from LifeScan Inc., in Milpitas, CA. Different temperature environments were simulated using several different chambers at Westpak Inc., in San Jose, CA. For the low temperature environment, a So-Low model SE-37-40 environmental freezer was used, while a Westpak aging chamber was used for the high temperature environment. The combination low/high temperature environment was created using a compact Tenney Jr. environmental chamber that cycled between the two temperature extremes. The smaller size of this chamber allowed for faster temperature equalization at both hot and cold extremes.

Procedure

Because few experiments have been conducted on this topic, and none using these types of discontinuous-thread caps and bottles were found in the literature, very little was known about how the experiment should be designed. Thus, a preliminary experiment was conducted over a relatively short time interval, using only ambient temperature conditions, in order to help design the main experiment. In the following section, the procedure for the preliminary experiment is described first, followed by that of the main experiment.

Preliminary Experiment Procedure

Empty bottles were secured, one at a time, into the digital torque meter. The caps were applied manually, and tightened to an application torque (T_A) value of 12 ±1 in.-lbs, as indicated by the meter. A custom-fitting chuck was used to ensure that neither the bottle nor the cap would become deformed during cap application (see Figure 2). The capped bottles were then left at ambient conditions for time intervals ranging from 15 minutes to four days. When the desired time had elapsed, the caps were manually removed using the same chuck, with the meter now recording removal torque (T_R). The same operator applied and removed all closures to reduce possible operator/instrument variability.

The recommended application torque for 28mm continuous-thread plastic caps is 12 in.-lbs [2]. To determine whether this application torque would also be a reasonable value for the discontinuous-thread samples to be used in this experiment, a small pre-test was performed. PP caps were applied to fifteen bottles, with five caps tightened to 12 ±1 in.-lbs, five more to 14 ±1 in.-lbs, and the remaining five to 16 ±1 in.-lbs. The caps were removed after approximately five minutes, and the bottle threads were carefully inspected using a Clausing model 4314 Optical Comparator. The bottles with caps applied at 16 in.-lbs had several marks visible on the threads, suggesting that the caps had been over-torqued. The bottles with caps applied at 14 in.-lbs had a few marks visible, though not on every bottle, and the bottles with caps applied at 12 in.-lbs had no apparent marks due to cap application. From this small test, it seemed that the threshold application torque value at which deformation began to occur was approximately 14 in.-lbs. Thus, it was decided that a lower application torque of 12 ±1 in.-lbs would be suitable.

Each sample group consisted of eight bottles: four with cross-linked HDPE caps and four with PP caps. Measured torque loss for each sample was simply the difference between application and removal torque, or $T_A – T_R$. Application torque varied slightly from one sample to the next, so a more useful quantity was percent torque loss (%R), given by the equation $\%R = 100x(T_A – T_R)/T_A$. This value was calculated for all samples, as were the means and standard deviations within each group of bottles with like caps. Time intervals used were 15 minutes, 30min, 1 hour, 24hrs, 48hrs, 72hrs and 96hrs. Thus, a single iteration of the experiment required 56 bottles: seven time intervals, each with eight bottles. The entire experiment was repeated three times, using a total of 168 bottles, to reduce the chance of poor data that might occur due to variations in a few bottles, operator error, or other factors. Again, all samples were exposed only to ambient temperature conditions throughout the preliminary experiment, so the only two independent variables were cap material and time.

Main Experiment Procedure

As in the preliminary experiment, empty bottles were secured, one at a time, into the same digital torque meter. The caps were applied manually, and tightened to an application torque (T_A) value of 12 ±1 in.-lbs, as indicated by the meter. As before, a custom-fitting chuck was used to ensure that neither the bottle nor the cap would become deformed during cap application (see Figure 2). The same operator applied and removed all closures to reduce possible operator/instrument variability. After caps were applied, the samples were divided into four groups, and each group was subjected to different temperatures, as shown in Table 1. Temperatures were used as defined by ASTM Standard D4332, Conditioning Containers, Packages, or Packaging Components for Testing [11].

For the low/high temperature mix group, samples were placed in a chamber that cycled between the high and low temperature extremes several times. The rate of temperature change (ΔT) when heating or cooling was approximately 1°C per minute, or 60°C per hour. The chamber was held at each temperature extreme for several hours to ensure that all bottles in the sample group had equilibrated. One complete cycle—from hot to cold, then back to hot—took approximately 12 hours, or half as long as the day-night-day cycle of a real-world distribution environment. The goal of the low/high mix environment was to determine whether this combination of heat and cold would have a greater effect on removal torque than only a single temperature extreme.

The bottles and caps were left in the different environmental conditions for time intervals of 3, 7, 14, and 28 days. The caps were then removed manually, with the meter now recording removal torque (T_R). Each sample group consisted of 30 bottles: 15 with cross-linked HDPE caps and 15 with PP caps. As before, torque loss [$T_A–T_R$] and percent torque loss [$\%R = 100x(T_A–T_R)/T_A$] were calculated, as were means and standard deviations within each group. With the addition of different temperatures, the main experiment had three independent variables: cap material, temperature, and time, as shown in Table 2. The full-factorial design of the experiment required a total of 480 bottles: 30 bottles per group, with a total of 16 groups (four different environments, each with four time intervals). This large sample size helped to minimize the chance of poor data due to variations in a few bottles, operator error, or other factors.

Results

Preliminary Experiment Results

As mentioned in the procedure section, mean percent torque reduction (%R) was recorded for each time interval of three different iterations; the average application and removal torque values for each replicate are listed in Table 3. The mean of these three replicates was also calculated, so that an overall trend in torque reduction over time could be observed. This mean of replicates of %R as a function of time is shown in Figure 3. The graph reveals several things. First, the rate of change (slope) in %R versus time was very similar for both material types. Second, both PP and cross-linked HDPE closures reached a steady state of torque loss after approximately 72 hours. Finally, it was found that the torque reduction was higher for PP (~ 56%) than for cross-linked HDPE caps (~ 40%). In other words, the PP caps were easier to remove, which was also subjectively apparent during testing.

Main Experiment Results

As with the preliminary experiment, mean percent torque loss (%R) was calculated for all samples, and for all time intervals; the average application and removal torque values for each time interval are listed in Table 4. Upon examination of the data, it was noted that within any specific group (i.e. – bottles with PP caps in the high temperature group), the percent torque loss changed very little over the observed time interval of 3 to 28 days. This result was somewhat by design: removal torque was only recorded after a minimum of three days, when an initial equilibrium had already been reached, as determined in the preliminary experiment. This lack of variation over time made it possible to remove the time variable from the equation. In other words, it was possible to pool the data from all four time intervals within each temperature group, and examine differences in torque reduction caused solely by exposure to different temperatures, and by different cap materials. Mean %R and standard deviation for each different temperature group, with data from all time intervals within each group pooled together, are shown in Figure 4.

This graph also reveals several things. First, it is apparent that torque reduction (%R) was higher for PP caps than for cross-linked HDPE caps for all temperature environments. In other words, the PP caps were easier to remove, just as in the preliminary experiment. This was once again subjectively apparent during testing. Second, when comparing %R between different temperature groups, it is immediately apparent that temperature had a significant effect on torque reduction. Both PP and cross-linked HDPE caps in the low temperature group experienced a smaller torque reduction than the control groups, while both groups exposed to high temperatures experienced a greater %R than control samples. In the low temperature environment, cross-linked HDPE caps had an average of 9.2% lower torque loss than controls, and PP caps had an average of 19% less than controls. In the high temperature group, torque loss increased 8% for cross-linked HDPE caps, and nearly 22% for PP caps compared to control samples.

Finally, it is interesting to note that samples in the low/high temperature mix group experienced nearly identical torque reduction to samples exposed only to high temperature: 51.2% vs. 52.6% for cross-linked HDPE caps, and 79.3% vs. 79.4% for PP caps. This suggests that torque reduction in the low/high mix-temperature samples was mostly affected by exposure to high temperature, and that the alternating low temperature cycles did little to counteract this “relaxing” of the closure seals. It should be noted that standard deviations for all groups were relatively low compared to actual %R values, indicating that the data is reliable. However, cross-linked HDPE caps had consistently higher standard deviations than PP caps. In other words, there was a greater variation in removal torques of cross-linked HDPE caps, or less predictability of off-torque values.

The data seem to support all of the research hypotheses, as a noticeable increase in torque loss was observed for both high temperature and mix groups, but not for the low temperature group. Also, the removal torque values between the high temperature group and mix group seemed to be nearly identical. To confirm these trends statistically, a directional (one-tailed) t-test was applied to each of the following combinations of data: high temperature group versus ambient, mix temperature group versus ambient, and high temperature group versus mix temperature group. The t-test had to be repeated twice for each comparison: once for bottles with cross-linked HDPE caps, and once for bottles with PP caps. The research hypotheses were confirmed to a high level of significance. High temperature and mix temperature groups both experienced greater torque loss than ambient groups, at a 99.9% level of significance ($p < .001$) for bottles with PP caps, and a 99% level of significance ($p < .01$) for bottles with cross-linked HDPE caps. When compared to each other, however, there was no significant difference between torque loss of high temperature and mix temperature groups.

The low temperature groups clearly did not have greater torque loss than ambient groups, as their mean %R values were consistently lower. To determine whether this difference was statistically significant, a directional t-test was applied in the opposite direction. It was found that low temperature groups experienced a statistically significant reduction in torque loss compared to ambient samples, with a 99.9% level of significance ($p < .001$) for bottles with PP caps, and a 99% level of significance ($p < .01$) for bottles with cross-linked HDPE caps.

Conclusions

The primary goal of this experiment was to compare torque loss for two different closure materials in a variety of temperature conditions. Clearly, temperature has a significant effect on removal torque for both PP and cross-linked HDPE caps. Samples exposed to low temperature had higher removal torque than ambient samples, possibly due to a hardening of both plastics that created more interlocking at a molecular level—in other words, the bottles and caps were "frozen" to each other. Samples exposed to high temperatures, on the other hand, experienced a significant reduction of removal torque, especially for the bottles with PP caps. The low/high mix environment had the same effect on removal torque as the high temperature environment.

The results obtained in this experiment can help to promote further related testing. Temperature is certainly one element of the distribution cycle, but every package system also experiences vehicle vibration, impacts due to loading and unloading, and many other factors that might affect removal torque. Researchers wishing to study the effects of any of these factors could use this procedure as a reference for setting up their own experiment. Data from this experiment could also serve as a reference for predicting how such additional factors might further affect torque loss. Additional experiments could prove useful to anyone working with similar bottles and caps, and to the packaging community as a whole.

Having the correct application torque—and subsequent removal torque—for a bottle cap is an important packaging consideration. To date, however, few experiments have been conducted using these types of bottles and caps. The data gathered in this experiment are very significant, because they can provide a better understanding of torque loss during prolonged exposure to high and low temperature extremes. These results can, in turn, be used to help make certain predictions about package performance in real-world distribution cycles, and to facilitate future experiments.

References

1. Nairn JF, Norpell TM. Closures, Bottle and Jar. In *The Wiley Encyclopedia of Packaging Technology*, Brody AL and Marsh KS (eds.); John Wiley & Sons: New York, 1997; 206-219.

2. Hanlon JF. *Handbook of Packaging Engineering*, Second Edition, Technomic Publishing Company, Inc., Lancaster PA, 1992.

3. American Society for Testing and Materials. Standard test methods for measurement of torque retention for packages with continuous thread closures, ASTM designation D 2063-91. 1991 (Reapproved 2002).

4. Soroka W. *Fundamentals of Packaging Technology*, Third Edition. Institute of Packaging Professionals: Naperville, IL, 2002.

5. Van Houten B. Bigger Growth Ahead for Medical Packaging, online article in *Pharmaceutical & Medical Packaging News*, August 2004.

6. Lockhart H. Determination of torque loss for a 28mm metal closure on plastic bottle. *School of Packaging, Michigan State University*. 1992.

7. Lai C-S, Greenway G. The effect of time on cap removal torque. In *Packaging Technology & Engineering*, June 1999; 16: 149-158.

8. Pisuchpen S. Model for predicting application torque and removal torque of a continuous thread closure. Masters thesis, Michigan State University. 2000; UMI Microform 1399664.

9. Pisuchpen S. Measuring and modeling the effect of time and temperature on removal torque and sealing force of a continuous thread closure. Bell & Howell Information and Learning Company: Ann Arbor, MI, 2002; UMI Microform 3092193.

10. Elboudwarej A. *Plastics, Composites and Glass Packaging* (SJSU course reader). Spartan Publications: San Jose, CA, 1997.

11. ASTM. Standard practice for conditioning containers, packages, or packaging components for testing, ASTM designation D 4332-01. 2001.

Table 1: Temperature conditions for sample testing

Sample Group	Temperature	Environment
Ambient (Control)	23 ± 1°C (73.4 ± 2°F)	Standard Conditioning
Low Temperature	-18 ± 2°C (0 ± 4°F)	Frozen Food Storage
High Temperature	60 ± 3°C (140 ± 6°F)	Desert
Low/High Temp. Mix	-18 ± 2°C to 60 ± 3°C	Frozen Storage / Desert

Table 2: Independent variables, main experiment

Independent Variable	Variable Level
Linerless Closure Type	HDPE (cross-linked), PP
Environment Temperature	-18, 23, 60, -18/60 degrees C
Duration of Exposure	3, 7, 14, 28 days

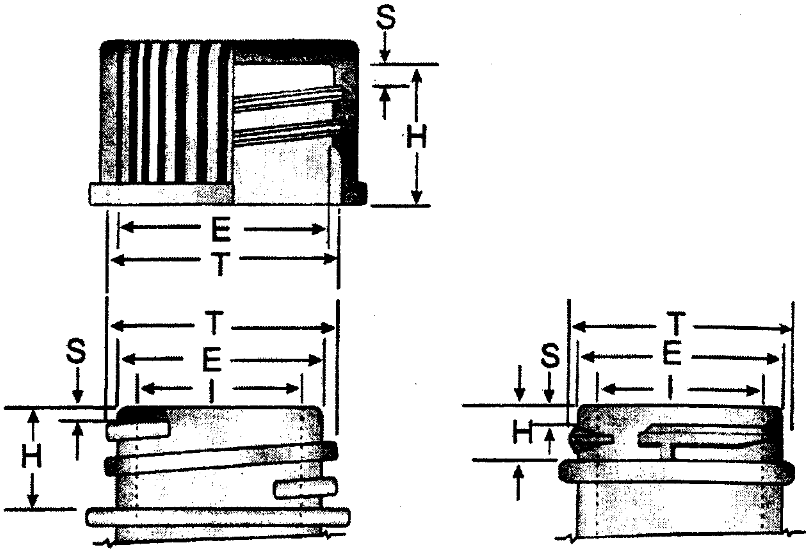
Table 3: Average and standard deviation application and removal torque measurements, preliminary experiment

		Iteration of Experiment					
		Replicate 1		Replicate 2		Replicate 3	
		Torque, in.-lbs. (Std. Dev.)		Torque, in.-lbs. (Std. Dev.)		Torque, in.-lbs. (Std. Dev.)	
Closure Type	Time (hours)	Application	Removal	Application	Removal	Application	Removal
HDPE	0.25	12.1 (0.2)	8.2 (0.9)	12.1 (0.2)	7.7 (1.4)	12.1 (0.2)	7.1 (1.1)
	0.5	12.0 (0.1)	6.8 (1.2)	12.0 (0.1)	7.2 (0.4)	12.2 (0.4)	7.3 (1.1)
	1	12.0 (0.1)	6.2 (0.9)	12.2 (0.4)	7.6 (1.0)	12.0 (0.1)	6.8 (1.8)
	24	11.9 (0.1)	5.8 (1.4)	12.2 (0.2)	6.0 (0.8)	12.1 (0.4)	6.2 (1.4)
	48	12.2 (0.5)	5.9 (1.6)	12.0 (0.1)	7.2 (0.7)	12.2 (0.2)	5.9 (1.1)
	72	12.1 (0.4)	7.0 (0.9)	11.9 (0.1)	7.6 (0.6)	12.0 (0.2)	7.0 (1.2)
	96	12.0 (0.2)	7.3 (0.7)	11.9 (0.2)	6.0 (2.6)	12.1 (0.4)	8.1 (0.3)
PP	0.25	12.0 (0.1)	7.9 (0.0)	11.9 (0.1)	8.3 (0.5)	12.0 (0.2)	7.2 (0.5)
	0.5	12.0 (0.1)	7.1 (0.5)	12.1 (0.1)	7.9 (0.1)	11.9 (0.1)	6.9 (0.3)
	1	12.1 (0.1)	7.0 (0.7)	12.1 (0.4)	8.0 (0.6)	12.0 (0.1)	6.7 (0.4)
	24	12.3 (0.4)	6.7 (0.9)	12.4 (0.4)	6.5 (0.1)	11.9 (0.1)	5.9 (0.5)
	48	12.0 (0.1)	6.3 (0.2)	12.1 (0.2)	6.6 (0.1)	12.0 (0.1)	6.1 (0.6)
	72	11.9 (0.1)	5.0 (0.2)	12.0 (0.2)	5.1 (0.2)	12.2 (0.3)	5.3 (0.1)
	96	11.9 (0.3)	5.4 (0.4)	11.9 (0.1)	5.4 (0.3)	11.9 (0.3)	5.5 (0.4)

Table 4: Average and standard deviation application and removal torque measurements, main experiment

		Duration of Exposure							
		3 days		7 days		14 days		28 days	
		Torque (Std Dev)		Torque (Std Dev)		Torque (Std Dev)		Torque (Std Dev)	
Cap Type	Group	T_A	T_R	T_A	T_R	T_A	T_R	T_A	T_R
HDPE	1	11.9 (0.5)	6.2 (1.4)	11.9 (0.4)	7.1 (1.0)	11.8 (0.5)	6.7 (1.8)	11.8 (0.7)	6.3 (1.5)
	2	11.8 (0.4)	7.6 (1.5)	11.8 (0.3)	7.7 (1.2)	11.6 (0.6)	7.6 (1.8)	12.2 (1.0)	7.7 (1.2)
	3	11.5 (0.4)	5.2 (0.7)	11.8 (0.5)	5.6 (1.4)	11.7 (0.4)	5.7 (1.0)	11.6 (0.3)	5.5 (1.2)
	4	11.8 (0.4)	5.9 (2.0)	12.1 (0.9)	6.0 (1.2)	11.9 (0.6)	5.7 (1.0)	11.9 (0.6)	5.8 (2.6)
PP	1	11.9 (0.4)	5.4 (0.3)	11.8 (0.4)	5.2 (0.3)	11.6 (0.6)	4.7 (0.5)	11.7 (0.5)	4.7 (0.3)
	2	11.8 (0.5)	7.6 (0.6)	11.5 (0.3)	7.0 (0.4)	11.9 (0.9)	7.3 (0.8)	11.6 (0.4)	7.1 (0.6)
	3	11.9 (0.6)	2.6 (0.3)	11.9 (0.6)	2.5 (0.3)	11.9 (0.5)	2.4 (0.2)	11.8 (1.0)	2.3 (0.5)
	4	11.7 (0.5)	2.6 (0.8)	11.9 (0.7)	2.5 (0.6)	11.9 (0.7)	2.5 (0.3)	11.9 (0.6)	2.3 (0.5)

Figure 1: Continuous thread (left) and lug style thread (right)



E	Thread root diameter.
I	Diameter at smallest opening inside finish.
H	Top of finish to top of bead or to intersection with bottle shoulder on beadless designs.
T	Thread diameter measured across the threads.
S	The vertical distance from the top of the finish to the start of the thread.

Figure 2: Cap being applied to bottle in torque meter

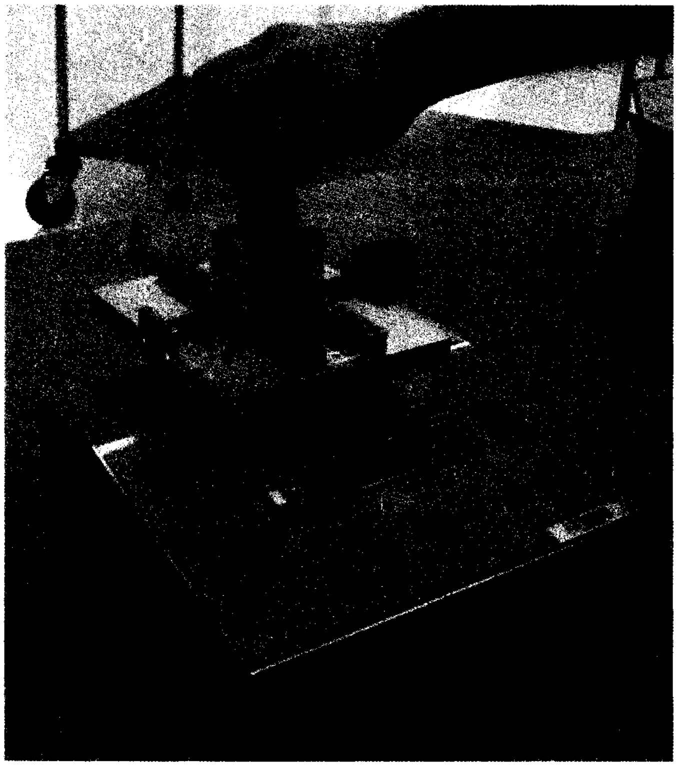


Figure 3: Torque loss of cross-linked HDPE vs. PP caps in preliminary experiment

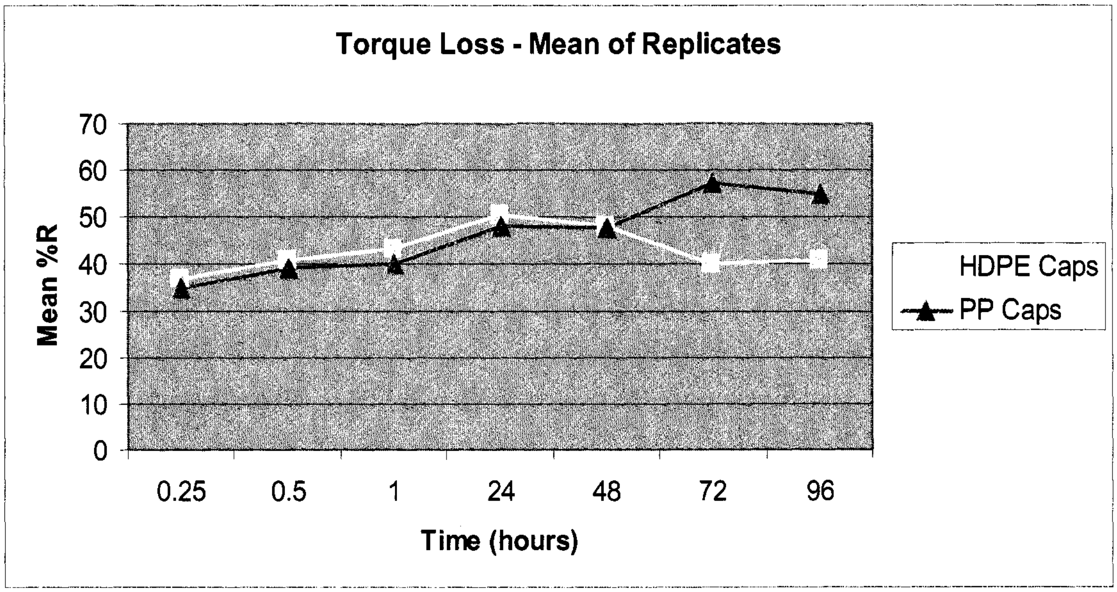
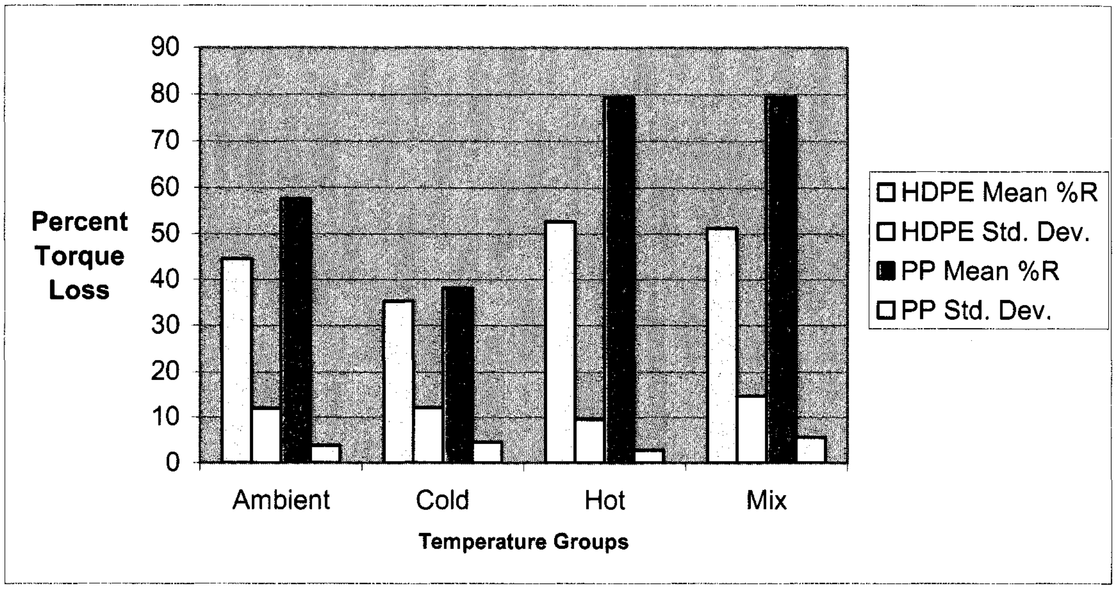


Figure 4: Torque loss means and standard deviations for pooled times



CHAPTER 3:

Summary and Recommendations

Summary

This research examined the effect of different temperatures on the removal torque of linerless discontinuous-thread PP and cross-linked HDPE closures (Figures 5 and 6), which were applied to 50ml HDPE bottles (Figures 7 and 8). The main piece of equipment used was a Kaps-All EB 550 digital torque meter. To achieve the desired high and low temperature extremes, several environmental chambers and monitoring devices at Westpak, Inc. were used, as shown in Table 5. For the low/high mix environment, the Tenney Jr. chamber was programmed to cycle between the low and high temperature extremes approximately every 12 hours, as shown in Figures 9 and 10.

Individual application and removal torques were measured for all temperature groups and time intervals, as shown in Tables 6 through 13. Mean torque losses for all groups with cross-linked HDPE caps are shown graphically in Figure 11, while mean %R values for all groups with PP caps are shown in Figure 12. It was noted that within any specific group (i.e. – bottles with PP caps in the high temperature group), the percent torque loss changed very little over the observed time interval of 3 to 28 days. This allowed data from all time intervals to be pooled, so that torque reduction caused solely by exposure to different temperatures and by different cap materials could be examined.

Temperature has a significant effect on removal torque for both PP and cross-linked HDPE caps. Samples exposed to low temperature had higher removal torque than ambient samples, possibly due to a hardening of both plastics that created more interlocking at a molecular level—in other words, the bottles and caps were “frozen” to each other. Samples exposed to high temperatures, on the other hand, experienced a significant reduction of removal torque, especially for the bottles with PP caps. The low/high mix environment had the same effect on removal torque as the high temperature environment.

Limitations and Recommendations

Temperature is certainly one element of the distribution cycle, but every package system also experiences vehicle vibration, impacts due to loading and unloading, and other factors that might affect removal torque. This experiment used only one type of bottle and two closures of the same design; having a more diverse group of samples would have likely produced additional useful data. As mentioned before, few prior studies in this area were found in a literature review. Since there was no model upon which to base this experiment, considerable effort went into a preliminary experiment to establish testing parameters.

It should be noted that the result obtained in this experiment is contrary to conventional wisdom among closure manufacturers, which states that using like materials for both the bottle and closure will cause more torque loss than using dissimilar materials. There are several possible reasons for this discrepancy.

Colorants or other additives might have caused a higher degree of static friction, or it could be a factor inherent in the thread design or pitch. It is also likely that the cross-linked HDPE caps in this experiment experienced less deformation than regular HDPE caps would have, and that this contributed to their torque retention. Further research in this area is warranted, as no other experiments have been conducted using these types of bottles and closures. Nonetheless, the data gathered in this experiment are very useful, because they can provide a better understanding of torque loss during prolonged exposure to high and low temperatures. These results can, in turn, be used to help make some predictions about package performance in real-world distribution cycles.

Bottles and closures made of HDPE and PP are popular because of excellent physical characteristics that allow them to be used in a wide range of applications, and because of their low cost. It should be noted, however, that these plastics are petroleum-based, so their costs tend to fluctuate with the petroleum market. For example, the price of crude oil rose by 80 percent during 2004, so the price of plastic pellets also rose accordingly (Spohr, 2004). A recent pricing report from Plastics Technology stated that both PP and polyethylene resin prices have increased from last year, and were expected to go up an additional 3¢ to 4¢ by the second quarter (Sherman and Schut, 2005). Cost is always a consideration when choosing a packaging material. If a less expensive material can offer comparable performance in a desired area, such as torque retention, then it will usually be the first choice for packaging manufacturers.

References

American Society for Testing and Materials (1991). *Standard Test Methods for Measurement of Torque Retention for Packages with Continuous Thread Closures,* ASTM designation D 2063-91 (Reapproved 2002).

ASTM (1997). *Standard Test Method for Application and Removal Torque of Threaded or Lug-Style Closures,* ASTM designation D 3198-97 (Reapproved 2002).

ASTM (2001). *Standard Practice for Conditioning Containers, Packages, or Packaging Components for Testing,* ASTM designation D 4332-01.

Elboudwarej, Albert (1997). *Plastics, Composites and Glass Packaging* (SJSU course reader). San Jose, CA: Spartan Publications.

Greenway, Gerald (1993). A short-term study of cap torque decay. *Packaging Technology and Engineering, May 1993,* pp. 38-39.

Hanlon, Joseph F. (1992). *Handbook of Packaging Engineering*, Second Edition. Lancaster PA: Technomic Publishing Company, Inc.

Khol, Ronald (1999). Stress analysis with creep is no easy matter. *Machine Design*, v71 no6 p44.

Lai, Ching-Sung and Greenway, Gerald (1999). The effect of time on cap removal torque. *Packging Technology & Engineering*, 6, 34-36.

Lockhart, Hugh (1992). *Determination of torque loss for a 28mm metal closure on plastic bottle*. School of Packaging, Michigan State University.

Nairn, J. F. & Norpell, T. M. (1997). Closures, bottle and jar. In A. Brody & K. Marsh (Eds.), *The Wiley Encyclopedia of Packaging Technology*, Second Edition (pp. 206-219). New York: John Wiley & Sons.

Pisuchpen, Supachai (2000). *Model for predicting application torque and removal torque of a continuous thread closure*. Ann Arbor, MI: Bell & Howell Information and Learning Company. UMI Microform 1399664.

Pisuchpen, Supachai (2002). *Measuring and modeling the effect of time and temperature on removal torque and sealing force of a continuous thread closure*. Ann Arbor, MI: Bell & Howell Information and Learning Company. UMI Microform 3092193.

Sherman, Lilli Manolis and Schut, Jan H. (2005). Pricing update – April 2005, Commodity prices rise more slowly. Retrieved May, 2005, from *Plastics Technology* website.

Soroka, Walter (2002). *Fundamentals of Packaging Technology*, Third Edition. Naperville, IL: Institute of Packaging Professionals.

Spohr, George (2004). Plastics manufacturers get 'lean and mean.' Retrieved March, 2005, from *Central New York Business Journal* website.

Van Houten, Ben (2004). Bigger Growth Ahead for Medical Packaging. Retrieved April, 2005, from *Pharmaceutical & Medical Packaging News* website.

Table 5: List of chambers and equipment used

Sample Group	Instrumentation Component	Westpak #	Model #	Calibration Date
All Temperature Groups	Kaps-All Digital Torque Meter	N/A	EB-550	May 2004
Low Temperature	So-Low environmental freezer	375	SE-37-40	Not Required
	Fenwal controller	376	400	07/12/2000
	Dickson Chart Recorder Temp Only	208	KTX	10/09/2002
High Temperature	Westpak Aging Chamber (Ringo)	239	TH4.5x4x3.5	Not Required
	Watlow Temperature and Humidity Controller	290	945	07/23/2002
	Honeywell Chart Recorder	315	DR45AT-1100-00-000-0-000P00-0	07/23/2002
Combination Temperature	Tenney JR.: Tenney temperature chamber	402	Tenney JR.	Not Required
	Watlow controller	346	942	07/17/2002
	Honeywell Chart Recorder	347	DR4500	07/17/2002
Thermocouples	Agilent Data Logger	420	34970A	12/09/2002

Table 6: Individual application torques, averages and standard deviations of ambient groups

Bottle Number	Cross-linked HDPE Closures				Polypropylene Closures			
	Duration of Exposure to Environmental Condition							
	3 Days	7 Days	14 Days	28 Days	3 Days	7 Days	14 Days	28 Days
1	11.6	11.5	12.2	12.0	11.8	11.8	11.4	11.2
2	11.9	11.7	11.5	11.8	11.6	11.7	11.4	12.7
3	11.7	12.3	11.3	11.5	11.3	12.0	11.5	11.5
4	11.7	11.6	12.1	11.3	11.7	11.9	11.4	11.4
5	12.4	11.6	12.0	11.6	12.2	11.9	11.0	11.2
6	12.0	12.1	11.7	11.3	11.9	12.5	11.9	11.8
7	11.6	11.9	11.8	11.3	11.6	11.5	11.4	12.3
8	11.9	12.8	11.9	12.6	11.6	11.4	13.0	11.6
9	11.9	11.9	11.6	12.0	12.0	11.4	11.2	11.5
10	11.7	11.6	12.0	11.3	11.8	12.1	11.2	11.6
11	12.3	12.0	12.5	14.0	11.7	11.5	11.8	11.2
12	11.5	12.0	12.9	11.7	11.6	11.7	11.2	11.6
13	11.7	12.5	11.3	11.5	11.7	12.0	11.2	11.9
14	11.3	11.4	11.1	11.8	12.7	11.0	13.0	12.0
15	13.2	11.7	11.7	11.5	12.6	12.4	11.4	12.4
Mean	11.9	11.9	11.8	11.8	11.9	11.8	11.6	11.7
Std. Dev.	0.5	0.4	0.5	0.7	0.4	0.4	0.6	0.5

Table 7: Individual application torques, averages and standard deviations of low temperature groups

Bottle Number	Cross-linked HDPE Closures				Polypropylene Closures			
	Duration of Exposure to Environmental Condition							
	3 Days	7 Days	14 Days	28 Days	3 Days	7 Days	14 Days	28 Days
1	12.1	11.9	11.2	11.0	12.5	11.4	11.3	11.9
2	11.9	12.1	12.5	11.2	11.5	11.2	11.1	11.9
3	11.7	11.6	11.2	11.1	12.1	11.3	11.3	11.2
4	12.4	11.8	11.6	13.0	11.5	11.8	11.1	11.4
5	11.5	11.2	11.3	11.7	11.5	11.7	11.5	11.6
6	11.8	11.7	11.3	11.7	11.7	12.1	11.8	11.2
7	11.8	11.8	11.1	11.3	11.5	11.7	11.9	11.8
8	11.8	11.3	11.9	11.6	12.3	11.5	11.3	11.1
9	11.4	12.2	11.5	12.0	11.4	11.5	12.0	11.3
10	11.4	12.4	11.4	14.6	12.3	11.2	11.3	11.4
11	11.5	12.1	11.0	13.9	12.9	12.3	11.3	11.6
12	11.4	12.2	13.4	12.1	11.3	11.5	11.7	12.2
13	12.7	11.7	11.9	13.0	11.1	11.5	12.9	11.2
14	11.9	11.6	11.3	12.4	11.9	11.1	13.7	12.6
15	11.3	11.5	11.5	12.3	11.6	11.1	13.9	11.8
Mean	11.8	11.8	11.6	12.2	11.8	11.5	11.9	11.6
Std. Dev.	0.4	0.3	0.6	1.0	0.5	0.3	0.9	0.4

Table 8: Individual application torques, averages and standard deviations of high temperature groups

Bottle Number	Cross-linked HDPE Closures				Polypropylene Closures			
	Duration of Exposure to Environmental Condition							
	3 Days	7 Days	14 Days	28 Days	3 Days	7 Days	14 Days	28 Days
1	11.9	11.3	11.2	11.6	12.0	11.3	11.2	14.9
2	11.0	11.6	11.2	11.3	11.8	12.9	11.5	11.1
3	11.6	11.4	11.6	11.3	11.1	11.4	12.5	11.1
4	11.6	12.2	12.4	11.4	12.2	11.6	11.2	12.4
5	11.4	11.6	11.6	11.7	11.3	11.6	12.2	11.1
6	11.7	11.0	12.0	11.4	11.3	13.0	11.5	11.0
7	11.3	11.9	11.9	11.7	11.2	11.8	11.3	11.2
8	11.2	11.6	11.8	12.4	11.7	11.8	13.2	11.2
9	11.4	13.1	12.1	11.8	12.4	12.8	11.8	12.5
10	12.3	12.0	11.3	11.1	11.8	11.3	11.5	11.8
11	11.3	12.1	11.4	11.6	13.5	11.8	12.0	11.0
12	12.0	12.0	11.0	11.2	11.4	11.6	11.9	11.4
13	11.6	11.4	12.4	11.7	11.6	11.9	12.0	12.4
14	11.1	11.7	11.6	11.6	11.9	11.7	12.0	12.6
15	11.3	11.6	11.6	12.1	12.6	12.7	12.0	11.6
Mean	11.5	11.8	11.7	11.6	11.9	11.9	11.9	11.8
Std. Dev.	0.4	0.5	0.4	0.3	0.6	0.6	0.5	1.0

Table 9: Individual application torques, averages and standard deviations of low/high temperature mix groups

Bottle Number	Cross-linked HDPE Closures				Polypropylene Closures			
	Duration of Exposure to Environmental Condition							
	3 Days	7 Days	14 Days	28 Days	3 Days	7 Days	14 Days	28 Days
1	11.5	11.9	12.2	13.5	12.0	11.9	11.4	11.2
2	12.5	13.0	11.8	11.3	11.7	11.8	11.4	12.7
3	12.0	11.6	12.0	11.8	11.8	11.5	11.2	12.6
4	11.1	11.3	13.0	11.6	12.5	11.1	11.7	12.3
5	11.7	12.4	11.3	11.8	11.5	11.7	12.2	12.0
6	11.7	11.2	11.3	12.9	11.1	13.0	11.2	11.4
7	11.3	12.0	11.4	11.7	11.0	11.8	12.7	11.1
8	12.0	12.4	11.5	11.8	11.3	12.8	12.0	11.2
9	12.1	12.0	11.3	11.8	12.1	11.1	11.3	12.7
10	11.6	11.7	11.9	11.5	11.4	12.9	12.3	11.5
11	11.8	15.0	13.2	11.9	11.9	11.5	11.9	12.5
12	11.7	12.0	12.7	11.2	11.5	11.7	11.4	11.2
13	11.4	12.3	12.4	11.9	12.4	11.0	11.3	12.7
14	12.5	11.3	11.6	11.8	12.2	12.8	13.4	11.9
15	12.5	11.5	11.6	12.2	11.5	12.5	12.5	11.4
Mean	11.8	12.1	11.9	11.9	11.7	11.9	11.9	11.9
Std. Dev.	0.4	0.9	0.6	0.6	0.5	0.7	0.7	0.6

Table 10: Individual removal torques, averages and standard deviations of ambient groups

Bottle Number	Cross-linked HDPE Closures				Polypropylene Closures			
	Duration of Exposure to Environmental Condition							
	3 Days	7 Days	14 Days	28 Days	3 Days	7 Days	14 Days	28 Days
1	6.8	6.3	7.6	8.0	5.5	5.1	4.2	5.2
2	7.1	8.4	9.4	6.8	5.3	4.8	4.7	4.6
3	6.4	8.3	6.0	7.6	5.8	4.9	4.8	5.0
4	5.6	5.4	8.4	6.0	5.3	5.3	4.5	5.1
5	5.4	8.0	4.1	7.7	5.2	5.5	4.1	4.4
6	3.4	8.0	6.0	6.6	5.4	5.1	4.4	4.6
7	5.6	5.9	6.7	3.7	5.1	5.4	4.3	4.2
8	6.3	6.7	7.6	8.5	4.8	4.9	5.1	4.5
9	6.7	8.0	7.5	6.5	5.3	5.2	4.7	5.1
10	7.4	6.5	7.2	6.8	4.9	5.7	4.6	4.2
11	6.5	7.3	9.2	5.6	5.6	5.3	5.8	4.7
12	7.4	7.7	5.1	7.7	5.6	4.9	4.6	4.6
13	5.0	5.8	6.1	4.8	5.2	5.6	4.6	4.6
14	4.3	6.8	2.9	5.6	5.5	5.4	5.5	4.4
15	9.0	6.9	6.8	3.3	5.8	5.5	4.5	4.6
Mean	6.2	7.1	6.7	6.3	5.4	5.2	4.7	4.7
Std. Dev.	1.4	1.0	1.8	1.5	0.3	0.3	0.5	0.3

Table 11: Individual removal torques, averages and standard deviations of low temperature groups

Bottle Number	Cross-linked HDPE Closures				Polypropylene Closures			
	Duration of Exposure to Environmental Condition							
	3 Days	7 Days	14 Days	28 Days	3 Days	7 Days	14 Days	28 Days
1	7.0	7.7	4.6	5.5	7.4	7.9	6.3	7.3
2	5.5	6.4	8.4	7.3	6.6	7.0	6.1	6.7
3	8.5	6.6	9.6	8.4	7.0	7.3	6.8	6.1
4	7.0	9.0	4.5	7.9	7.5	6.6	6.4	6.5
5	9.3	5.3	6.1	5.9	7.5	6.5	7.4	6.5
6	6.8	8.4	8.6	8.8	6.9	7.4	7.1	7.2
7	4.5	6.2	9.9	8.0	8.1	6.8	7.2	7.1
8	6.3	8.1	5.2	7.3	7.7	6.3	7.2	6.5
9	8.6	8.1	9.0	9.5	7.7	6.8	6.9	6.7
10	7.1	6.6	7.2	9.3	8.7	6.6	7.7	7.2
11	8.4	8.5	7.7	8.5	7.4	7.6	7.2	7.5
12	7.9	9.0	9.2	5.9	7.7	6.8	7.3	7.8
13	7.9	8.8	7.6	7.8	7.0	6.7	7.9	7.9
14	9.9	6.9	8.5	7.2	8.5	7.2	9.0	8.4
15	9.0	9.3	8.5	8.0	8.1	7.1	8.5	6.9
Mean	7.6	7.7	7.6	7.7	7.6	7.0	7.3	7.1
Std. Dev.	1.5	1.2	1.8	1.2	0.6	0.4	0.8	0.6

Table 12: Individual removal torques, averages and standard deviations of high temperature groups

Bottle Number	Cross-linked HDPE Closures				Polypropylene Closures			
	Duration of Exposure to Environmental Condition							
	3 Days	7 Days	14 Days	28 Days	3 Days	7 Days	14 Days	28 Days
1	5.7	5.7	6.7	5.8	2.8	1.8	2.5	2.4
2	4.8	6.4	6.4	4.6	2.4	2.8	2.5	1.8
3	4.4	6.7	3.1	6.5	2.2	2.4	2.2	1.7
4	5.8	6.0	5.3	5.2	2.5	2.5	2.7	3.3
5	5.9	6.3	6.0	5.4	1.9	3.0	1.9	1.7
6	5.3	5.1	4.5	6.5	2.3	2.6	2.5	2.2
7	5.3	2.3	6.3	6.2	2.4	2.0	2.2	2.4
8	3.9	5.6	5.5	6.2	2.7	2.5	2.3	1.7
9	0.0	6.5	6.8	3.4	2.7	2.4	2.6	2.3
10	6.2	2.9	5.8	7.4	2.7	2.7	2.4	2.7
11	4.5	6.7	0.0	2.7	2.8	2.8	2.6	2.7
12	4.7	5.4	5.5	5.8	2.7	2.3	2.3	2.3
13	5.4	6.0	5.6	6.0	2.7	2.4	2.0	2.3
14	5.0	0.0	6.6	5.7	2.7	2.4	2.7	2.7
15	6.5	6.7	5.9	4.8	2.8	3.0	2.3	2.8
Mean	5.2	5.6	5.7	5.5	2.6	2.5	2.4	2.3
Std. Dev.	0.7	1.4	1.0	1.2	0.3	0.3	0.2	0.5

Table 13: Individual removal torques, averages and standard deviations of low/high temperature mix groups

Bottle Number	Cross-linked HDPE Closures				Polypropylene Closures			
	Duration of Exposure to Environmental Condition							
	3 Days	7 Days	14 Days	28 Days	3 Days	7 Days	14 Days	28 Days
1	2.0	3.2	3.3	8.0	1.6	1.9	2.0	0.0
2	10.2	7.3	6.0	2.9	1.7	1.8	3.0	1.7
3	3.6	5.5	6.4	6.9	1.9	3.3	2.3	2.1
4	6.4	6.2	6.4	2.3	1.8	2.5	2.3	1.7
5	5.6	5.8	5.9	7.2	1.7	1.9	2.4	2.4
6	6.1	6.6	4.6	7.5	3.0	2.8	2.8	2.3
7	5.4	0.0	5.5	3.6	3.3	2.6	2.5	2.3
8	7.7	6.2	4.9	8.1	2.1	4.1	2.3	2.3
9	5.8	6.4	6.2	7.5	3.7	3.3	2.4	2.2
10	5.6	6.0	4.4	7.5	3.1	2.2	3.0	1.9
11	4.3	7.7	6.7	2.2	2.2	2.7	3.1	2.7
12	5.4	6.4	5.8	2.4	3.1	1.9	2.6	2.4
13	7.9	7.0	6.1	9.5	2.9	2.4	2.5	3.6
14	8.1	0.0	0.0	8.1	3.9	2.5	2.5	2.1
15	4.3	4.0	7.0	3.9	2.5	2.3	1.9	2.7
Mean	5.9	6.0	5.7	5.8	2.6	2.5	2.5	2.3
Std. Dev.	2.0	1.2	1.0	2.6	0.8	0.6	0.3	0.5

Figure 5: Cross-section of cross-linked HDPE linerless closure

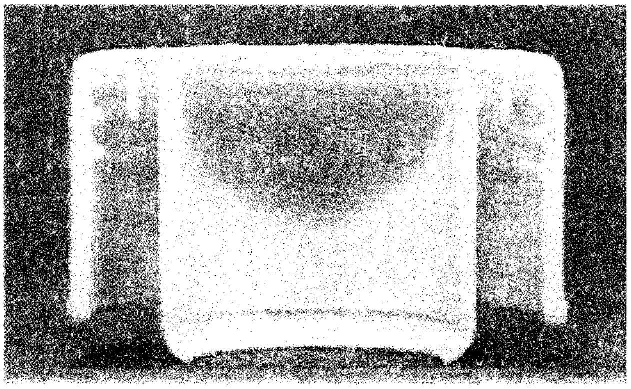


Figure 6: Cross-section of PP linerless closure

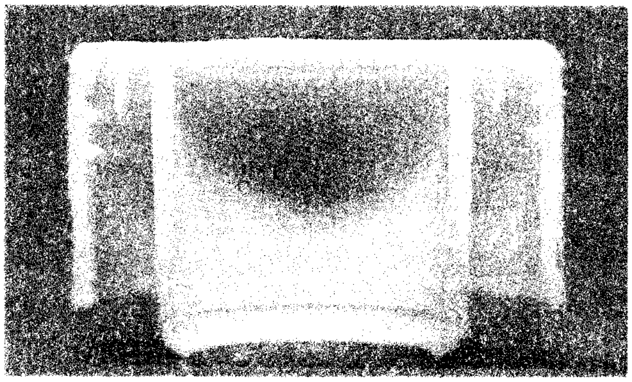


Figure 7: HDPE 50ml bottle with 1/3-turn discontinuous thread

Figure 8: Bottles with cross-linked HDPE closure (left) and PP closure (right)

Figure 9: Chamber temperature for hot/cold mix group, first week

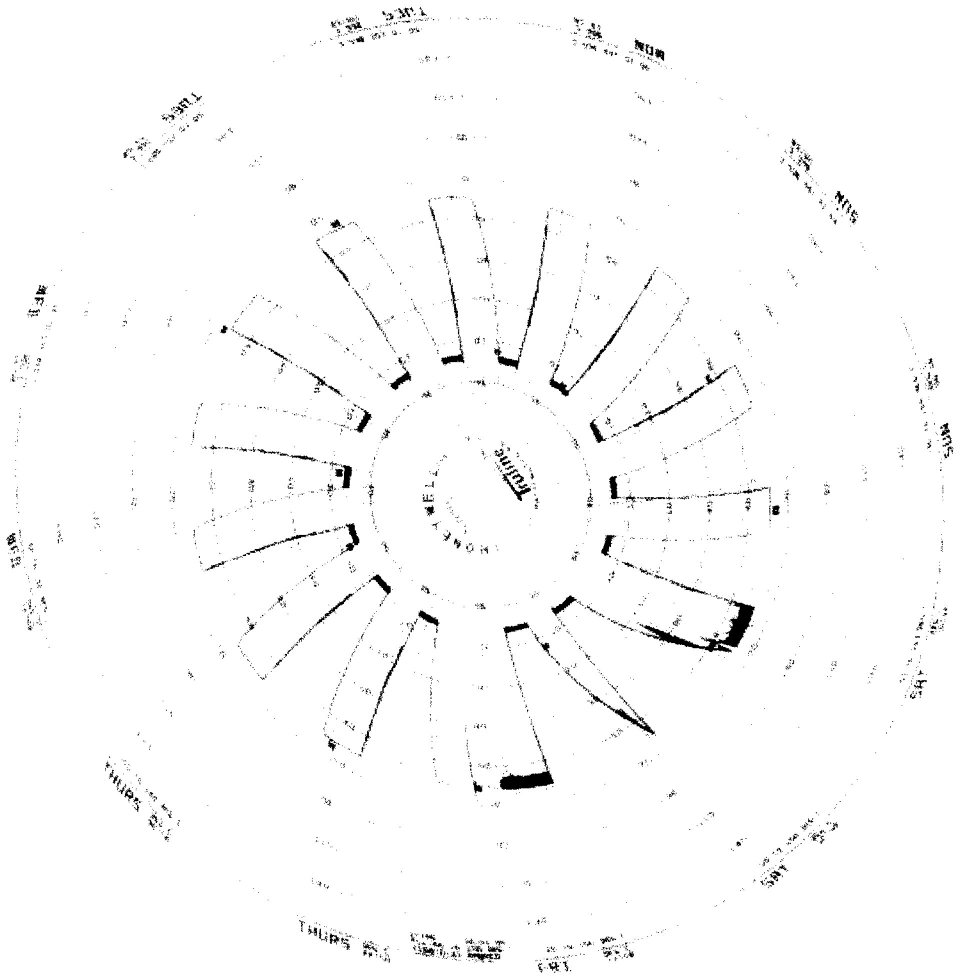


Figure 10: Chamber temperature for hot/cold mix group, weeks 2-4

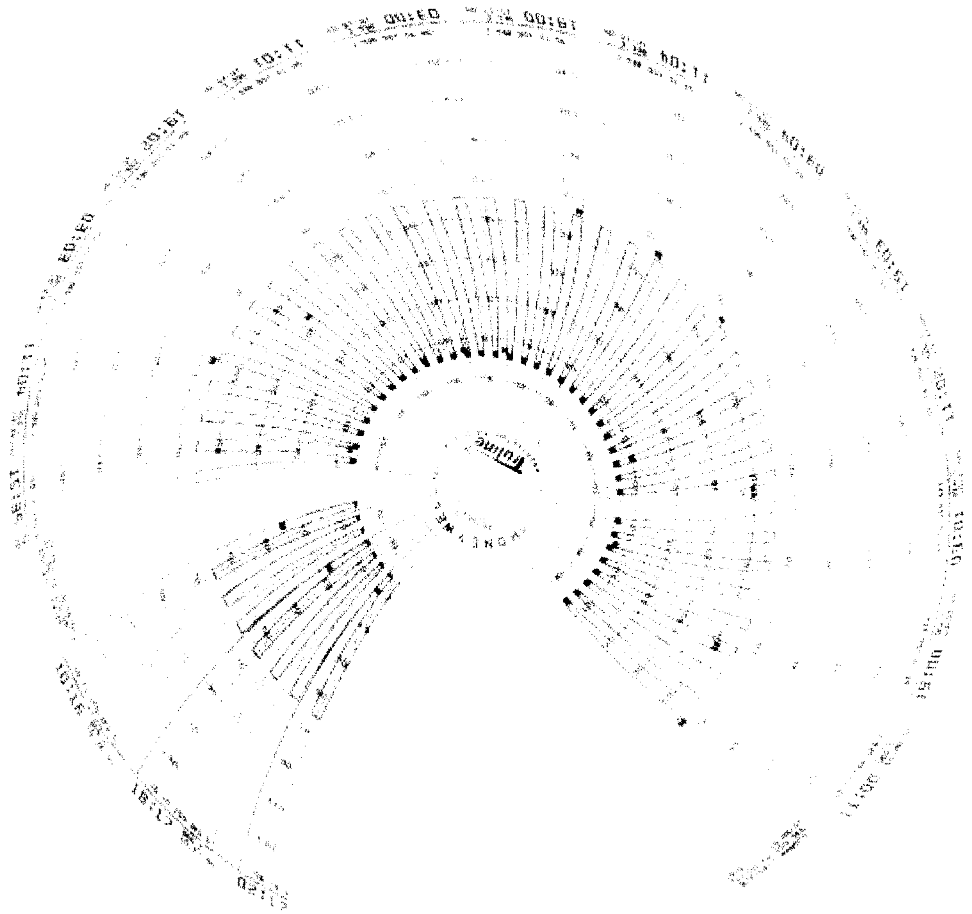


Figure 11: Torque loss of temperature groups with cross-linked HDPE caps

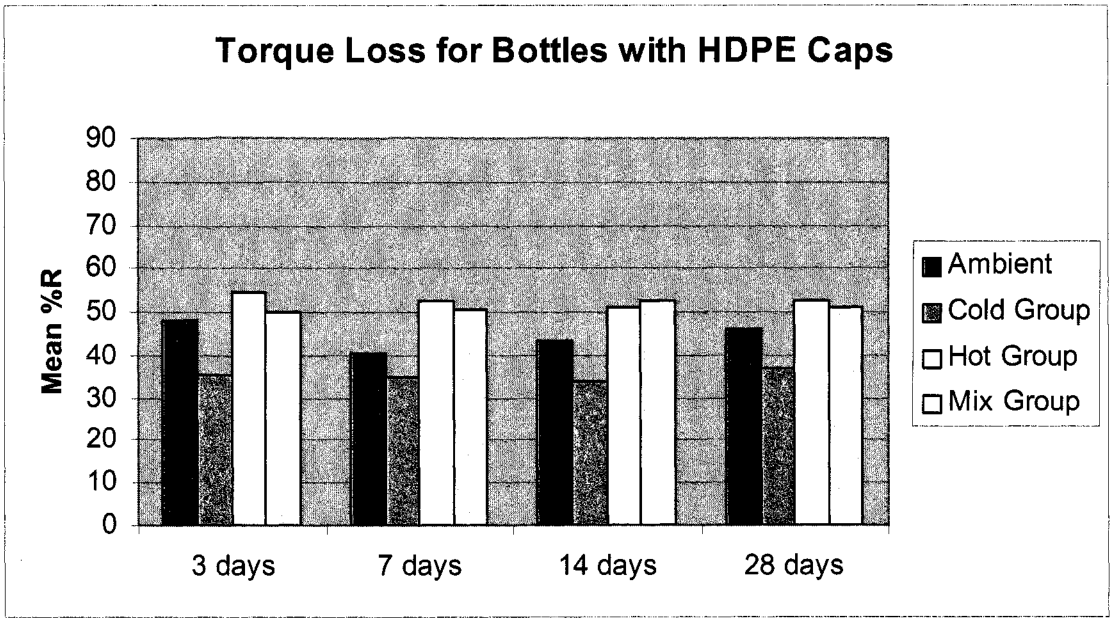


Figure 12: Torque loss of temperature groups with PP caps