This is to certify that the

dissertation entitled

LEAKERS IN GALLON SIZE PLASTIC BOTTLES
IN SIMULATED SMALL PARCEL TEST ENVIRONMENT

presented by

VIVEK PRAKASH SYAL

has been accepted towards fulfillment
of the requirements for

MASTER degree in PACKAGING

S. Paul Singh
Major professor

Date DECEMBER 12, 1994

LEAKERS IN GALLON SIZE PLASTIC BOTTLES IN SIMULATED SMALL PARCEL TEST ENVIRONMENT

Vivek Prakash Syal

A Thesis

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

Master of Science

School of Packaging

1994

ABSTRACT

This study investigated the effect of vibration and drops on gallon size plastic bottles when subjected to simulated small parcel shipping tests. The three types of bottles were:

1. 33-400 finish, Round, gallon size, HDPE, 110 gram bottle.
2. 38-400 finish, Round, gallon size, HDPE, 110 gram bottle.
3. 63-400 finish, F-style, gallon size, HDPE, 125 gram bottle.

Three different types of closures were used on each of the bottle types described above. These were: Sure-Seal 200 series (foamed LDPE core with top/bottom layers of 2 mil LDPE), Foil-Seal 3/19 (heat induction foil lined), and the Tekni-Seal closure (pressure sensitive adhesive lined closure).

Six bottles of each type were filled with water and capped with each closure type and packaged in single wall RSC corrugated boxes with separators. The packages were subjected to the drop and vibration tests after being conditioned at both normal conditions of 72°F and 50% Relative Humidity and accelerated conditions of 72°F and 50% Relative Humidity for 72 hours. The bottles were checked for both application torques before testing and removal torques after testing. The results showed that the Sure-Seal type of closure showed the maximum number of leaks. Also the larger finish 63-400 F-style bottle showed the most leaks whereas the smaller finish 33-400 style round bottles showed the least number of leaks. The bottles that were subjected to the higher temperature conditioning showed more leaks and a greater reduction in removal torques.

This thesis is dedicated to my wife,

Indu, for all the love and support she has provided.

ACKNOWLEDGEMENTS

I would like to express my deepest appreciation to my major professor, Dr. S. Paul Singh for his assistance, guidance and support throughout my graduate study and research.

I would also like to extend my appreciation for the recommendations and support provided by the members of my committee, Dr. Gary J. Burgess and Dr. Galen K. Brown.

Finally, I would like to thank all my colleagues who helped me and encouraged me during my research project and the Consortium of Distribution Packaging for the funding.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF TABLES (continued)

LIST OF FIGURES

1.0 INTRODUCTION

The smallest part of a package, and often the most critical, is usually the closure. The security of the package assembly and the integrity of the components are dependent on the closure which may be a cap or tie. The closure has to maintain integrity during the assembly, storage, handling, and shipping. It must also be easy to open and re-close when it reaches the end consumer. Often there is a fine line between a closure that is easy to open and strong enough to travel, and one that fails in shipment or is nearly impossible for the average person to open.

In addition to its functional qualities, a closure can sometimes enhance the appearance of a package. A ribbon bow, an embossed metallized seal, or a decorative cap can provide the extra touch that makes the difference between success and failure in the marketplace. A message or brand identification can be included in the design of a closure, where it is most likely to be noticed. A closure is the finishing touch to the package, and it deserves special attention if it is to make the most of its prominent position (Hanlon, 1992).

1.1 HISTORICAL BACKGROUND.

The human civilization has been using closures for various package types over the past few centuries to preserve the contents from spilling and contamination. The Roman Empire has antecedents of high usage of glass bottles and jars. The use of cork floats and buoys by the Romans, with subsequent applications as bungs (large stoppers) for casks suggests the likelihood of the origin of fabricated cork bottle stoppers. Other sealing methods used in different parts of the world include a Near Eastern method of covering the container with interlaced strands of glass, or strips of linen and applying a secondary seal of pitch. Western Europeans used glass stopples and various lids of glass and clay before the common use of cork. Wax was another common closure type that was inserted into the neck and covered with leather or parchment. Raw cotton or wool, sometimes dipped in wax or rosin, was also used after being covered with parchment or sized cloth, and was then bound to the neck. Out of all the above, cork provided the best sealing characteristics. Cork has high cellular density, is incompressible, elastic, highly impervious to air and water penetration, and low in thermal conductivity, thereby making it an ideal choice for a closure material. By the mid-19th century, cork was the predominant closure, providing a friction seal for a variety of products (McKearin and Wilson, 1978).

Attempts were made during this era to introduce the concept of a threaded closure to the industry. This evolution began with John Mason's 1858 patent of the "mason jar" re-designed glass threads to accomplish a tighter and more dependable seal. This design has the thread starting a little below the top surface and eventually fading away before reaching the shoulder.

Previously the top edge often broke away and the bottom would jam the cap thread before the cap was all the way down (Hanlon, 1992).

The next key development was the crown cap, invented by William Painter of Baltimore. The Phoenix cap, a flat disk with a separate split ring to draw it down tight, was introduced in the 48, 53, 58 and 63 mm sizes. This closure was popular among food packers because it provided a hermetic seal.

In the early 1900's automation in closing of jars and bottles took place which created a demand for inexpensive, easy to use standardized closures. The crown cap seemed to be the solution for the beverage industry. Some plastic closures were used in the early 1920's for toothpaste tubes, but it was only when the price of bakelite phenolic was reasonable that they came into general use. Generally, more expensive than metal caps, plastic closures are used mostly for luxury items. The advantage of the freedom of design with a variety of colors and textures makes them very desirable for premium products. Nearly 10 billion plastic closures are used annually by the packaging industry.

Shortly after World War 1, glass and cap manufacturers, through the Glass Container Association, designed and standardized the shallow continuous thread cap (Lief, 1965).

1.2 BASIC FUNCTIONS OF A CLOSURE.

A closure is an access-and-seal device which attaches to various types of glass, plastic, paperboard and metal containers. These include tubes, vials,

bottles, cans, jars, tumblers, etc. The closure along with the container fulfills three primary functions:

1. To provide protective containment through a positive seal
2. To provide access and re-sealability according to various requirements of convenience and control, and
3. To provide a vehicle for visual, audible, and tactile, communication.

Each of the three functions are briefly reviewed in this section. Effective sealing and protective containment are achieved when the closure and container are integrated to form a unified protection system for the product during its use. It is essentially a two-way process where the product and its contents cannot escape, and there is a barrier against intrusion of gases, moisture vapor, and other contaminates.

A positive seal is attained when the contact points of the closure and the top of the container are pressed together to form a seal. Sometimes a resilient lining material is present which is compressed between the closure and the container, providing a tighter and secure seal. Liners could be made of paper, plastic, metal foil, or a composite material.

Two types of closure methods are used to provide containment and sealing. These are the friction fitting closures and thread engagement closures. A positive seal depends on the type of product, closure, container, seal desired, resiliency of the liner, flatness of the sealing surface, and the tightness or torque with which the closure is applied. Current closure designs are based on the demands of the marketplace where the consumer plays a very important role. There are certain legal mandates for access control. Many packages are ergonomically designed for easy opening, dispensing, and critical access control. The technology in closure design has always strived to provide tight seal with easy access but today's demands for the two, i.e. access control and easy access are so opposite that it becomes difficult to combine and eventually satisfy the two (Tang, 1984).

Two types of access controls are used by the industry. These are:

- Tamper evident closures
- Child resistant closures

The FDA has stated eleven options for making a package tamper evident, two of which apply to closures.

The closure also provides a prime position for communications, an integral part of today's packaging. Since the closure is handled and seen by the consumer every time the product is used, communication on the closure is an ideal feature often used by cosmetics and beverage bottlers.

1.3 METHODS OF CLOSURE.

Removable closures attach to containers using either thread engagement or friction engagement. The characteristics of these two types are described in this section. There are three basic kinds of thread engagement closures. These are continuous thread (CT), lug, and roll-on caps. The CT designs attain a seal through the attachment of a continuously threaded closure to a compatible threaded container neck. The lug cap has a shorter thread design which leads to access and re-closure being accomplished in one quarter turn. The roll-on is basically a blank un-threaded metal shell which becomes a closure when compressed to conform to the finish of a bottle.

Many bottles are sealed with simple metal or plastic closures that are pressed onto the top of the same and held in place by friction. The four basic kinds of friction fit closures are crowns, snap-fit caps, press-on caps, and stoppers.

However, as defined by their utility, the four classes of modern day closures are containment, convenience, control, and special purpose.

Containment Closures.

Though all kinds of closures provide containment, a containment closure is defined as a one piece cap whose primary function is to provide containment and access on vast production scales. Continuous thread caps, crowns, and roll-on's are described by this category of closures.

Convenience Closures.

The technology development in this field has basically been propelled by the customer demand in this area. Convenience closures provide ready access to liquids, powders, flakes, and granules for products that are poured, squeezed, sprinkled, sprayed, or pumped from their containers. The five kinds of convenience closures are spout, plug-orifice, applicator, dispensing-fitment, and spray and pump types.

Sealing Systems.

Though the smallest aspect of a package, the seal is responsible for keeping the entire concept intact. If the seal is not maintained by the closure, the product will loose its use life. The sub-parts of a sealing system can be liners and inner-seals. The lining material can be either a single substance (paperboard or thermoplastic) or a composite material. Synthetic thermoplastic liners include foamed and solid plastics of varying densities. A composite lining material consists of a backing and a facing. The backing, usually made of cellulose or thermoplastic, is designed to provide the proper compressibility to affect the seal and proper resiliency for resealing. Facing materials are numerous, as are the variables of products with which they have to contend. Examples are thermoplastic resin coated papers, laminated papers of foil or film or multi-layers devised for special applications. The inner-seal seals the mouth of the container flush with the liner. Common kinds of inner-seals are waxed pulp, pressure sensitive, and heat induction. The waxed pulp inner-seal is quite common with the food industry. After the filling operation, the container runs under a roller system which applies an adhesive to the lip of the container and then the cap is applied. On removal, the glassine adheres to the container while the pulp backing remains in the closure. Pressure sensitive inner-seals are generally foamed polystyrene. The heat-induction kind are plastic coated aluminum foils, often adhered to a waxed pulp base liner. After the cap is applied, the container passes under an electromagnetic field which causes the aluminum to generate heat. The plastic facing on the aluminum subsequently melts and adheres to the container (Phoenix Closures, 1984).

There are also some kind of plastic liner-less closures which provide a positive seal in certain circumstances, obviating the need for intermediary materials and secondary post liner operations.

1.4 CLOSURE MATERIALS

Nearly all the closures in today's market are made of plastic, metal, or glass.

Plastics.

These can be further subdivided into the thermosets (phenolic resins and urea compounds) and the thermoplastics (polyethylene, polypropylene etc). The thermoplastic materials can be softened once they are heated and the thermoset materials do not change once they are molded.

In general, thermoplastics offer lower weight, versatility of design, high chemical resistance to a wide variety of products, and economical resins and manufacturing processes. Their flexibility is important for contemporary closure design with emphasis on convenience and control devices. Thermoplastics offer good application and removal torques. They maintain a good seal and tend to resist back-off. Unlike thermosets, thermoplastics can be pigmented in all the colors. Most of these closures are made by injection molding although some are made by thermoforming. Polypropylene and Polyethylene account for the majority of all thermoplastic closures. Polypropylene has high resistance to stress cracking which is a basic for hinged closures. In fact, in thin hinged sections it tends to strengthen with use. It has excellent resistance to acids, alkalies, oils and greases at normal operating temperatures. Of the polyolefins it has the best resistance to heat but it becomes brittle at low temperatures.

Low Density Polyethylene (LDPE) is resilient and flexible, tasteless and odorless. It provides great moisture protection but is not a good gas barrier. The injection molding times are relatively short. High Density Polyethylene (HDPE) is stiffer, harder, and more impermeable than LDPE. Unless specially formulated, it will stress crack in the presence of some detergents. A particular drawback of HDPE is its potential for warpage and loss of torque.

Polystyrene is used for a very small percentage of the closures today. The homopolymer is attacked by many chemicals, is very brittle, has relatively low heat resistance and does not provide a good barrier against moisture or gases.

Thermosets (phenolics and urea compounds) have a wide range of chemical compatibility and temperature tolerances. Some of them can attain sub-zero temperatures whereas others can survive at temperatures higher than 300°F. The density and rigidity of the thermosetting plastics gives the material its heavy weight. Cycle time for thermosets is longer than thermoplastics, depending upon the thickness of the product and additives.

Phenolics are hard and dense. They are the stiffest of all plastics but are relatively brittle and low in impact strength. The properties depend a lot on the filler material used. The heat resistance of these materials is outstanding. Phenolics cost less than ureas and are easier to fabricate but are limited to black and brown colors.

Urea formaldehyde is one of the oldest plastic packaging materials, used since the early 1900's. The resin produces extremely hard, rigid closures with excellent dimensional stability. It has the highest mar resistance but it is also the most brittle. They are not affected by organic solvents but are affected by alkalies and strong acids. Urea compounds also do not build up static electricity, which leaves them free of dust (Hanlon, 1992).

Metals.

These are probably the strongest of all closures, good for nearly all kinds of applications. There are two types of steel closure materials: tinplate and tinfree steel. Tinplate closures are plated steel with a thin coating of tin on both sides that helps protect the base steel from rust and corrosion. Tin-free steel is now promising to become the dominant steel closure material. Crown closures are also made of tin-free steel.

Aluminum closures offer some great advantages among metal closures since they are lightweight, malleable, and resistant to atmospheric corrosion.

Glass.

Glass stoppers are used in commercial glassware and more so in cosmetic and fragrance packaging. A polyethylene base cap assists in friction-fitting the stopper into the bottle. Stoppers for the luxury product lines mostly have these kind of closures (Hanlon, 1992).

1.5 CLOSURE SELECTION.

A very basic guideline is presented for the selection of an ideal closure system. The 5 Cs of closures are:

a. Containment. Essential requirements of containment are product compatibility and the ability to provide functional protection. This objective has to be reached after careful evaluation of the choices available in terms of methods, type, materials, and sealing systems. Decisions may hinge upon whether to use a lining material or a liner-less closure. Other important variables to be taken note of are interaction of closure and container system and how they affect the efficacy of engagement and seal. Torque considerations also have to account for seal pressure (the amount of pressure exerted on the sealing surfaces), and strip torque (the torque at which a closure slips over the container threads).

b. Convenience. Opportunities for convenient dispensing started with a closure that provided a reduced number of turns, or broader "knurls" on the walls of the closure skirt to ensure opening and closing. Other options could be simple spouts, plug-orifice snap caps, and sprayers and pumps. Various other factors such as requirements of containment, type of sealing system, and the premium placed on convenience will determine options in dispensing closures.

c. Control. Some kinds of products are mandated by law to be packaged with a tamper evident or child resistant closure. Cost and sealing needs will determine options in control closures, as well as whether secondary sealing systems are required. Most of the consumer complaints arise from inadequate opening and closing of product containers. A very careful review and testing of control closure and lining system can prevent potential access problems with elderly or handicapped consumers.

d. Communications. Shelf appearance of a closure is seen as a reflection of product quality as it communicates this with a specific style and form. Sometimes it may even have information on the ingredients of the product. Closures sometimes communicate not only visually, but audibly as well. Steel vacuum button closures "pop" on opening to confirm the freshness of a product, and polypropylene plug-orifices "snap" when the seal is engaged. Determination of all these factors also contributes to the final form of closure design.

d. Cost. There has to be an optimal trade off between the actual requirements of the closure and what cost factors dictate. Again as an example, thermoplastic-mold costs may be more expensive than those for thermosets, but faster cycle times and cheaper resins may prove economical in the larger volume runs. Customized closure might be perceived as costly in the initial design stages, but once economies of scale are achieved, they might turn out to be economical.

1.6 CLOSURE SPECIFICATION.

A closure is specified by a series of numbers and or letters. For example a closure 33 - 400 will be one where the first number 33 refers to the inside diameter of the closure as measured in millimeters. Common closure diameters range from 22 -120 mm. The second set of numbers, 400 refers to the finish designation. The "finish" of a closure is the thread design, size, pitch, profile, length, and thickness of the engagement threads on plastic and metal closures and containers. Designations for the most common CT closures are 400 and 425 for shallow continuous thread designs, 410 for medium CT's and 415 for tall CT's. Today there are approximately a hundred finishes specified for a variety of glass containers.

With the introduction of the plastic bottles, it was realized that the threads on the plastic bottles are not the same as in a glass bottle. It does not provide accurate closure centering on the finish nor does it permit higher capping torques required to provide a positive seal on plastic containers. Therefore the divisional subcommittee of the Society of Plastics Industry developed specific finish dimensions, tolerance, and thread contours for blown plastic bottles. The two basic contours are the M - style and the L - style. A typical glass thread is rounded in contour. The M - style thread engaging surfaces are angled at 10 degrees and the L - style is angled at 30 degrees. Both the contours increase sealing abilities for closures on plastics bottles.

Four critical closure dimension are T, E, H, and S. The T dimension is the size of the root of the thread inside the closure. E is the inside dimension of the thread in the closure. H is the measurement from the inside top of the closure to the bottom of the closure skirt. S is the vertical dimension from the inside top of the closure to the starting point of the thread. These are shown in Figure 1. These critical closure dimensions and tolerances for metal and plastic closures designed for glass and plastic containers are represented in the voluntary standards for closure as issued by Closure Manufacturers' Association (McLean, 1984).

1.7 CLOSURE TRENDS.

The technology for closures has changed so rapidly in the past few years, as compared to the last fifty, that it is really surprising. The real driving force has been the consumer. A high premium is paid for convenience. At times the whole industry has realized the shortcomings of a particular kind of design and has redefined access control.

The consumer today has an increased pace of life and thus requires a lot of convenience items in his day to day activities. The dispensing closure is seen as an integral part of the total lifestyle. The convenience closures are both time and labor saving. They prevent spills, leaks and drips. Other functional trends which seem to dominate today's market place are larger closure sizes, increased use of stock mold caps to decrease private mold costs, and new concepts in liner-less closure design. Since innovative packaging can increase market shares, special emphasis is placed on improved tamper evidence, child resistance and convenience designs. These functions are becoming more and more integrated into one cap.

Over the years plastic closures have seen an increase in use. Some industry executives strongly believe that the entire industry will shift to plastic closures simply because plastics provide an opportunity to use lightweight containers, simplify capping, and improve color matching. The ductility of plastics accounts for the fast progress of plastic closures (U.S. Dept. of Commerce, 1985).

1.8 Study Objectives.

The purpose of this study was to investigate leakers in gallon size plastic bottles when they are subjected to the small parcel environment. In addition the effect of creep in reduction of torque was studied.

The project was funded by the Consortium of Distribution Packaging. Currently a large number of liquid products which include chemicals are shipped in gallon size HDPE plastic bottles using small parcel shipping companies such as Federal Express and United Parcel Service. This study investigated three different types of bottles and three different closure methods. The bottles were subjected to drop testing and vibration testing as recommended in the NSTA Project 1A. The initial application and final retention torques on the closures were measured after subjecting the bottles to these various tests.

2.0 EXPERIMENTAL DESIGN

The various types of bottles and closures used in this study and the test methods used are discussed in this chapter.

2.1 Bottle Types:

Three different types of bottles were selected in this study. They represent the majority of bottle types used in the small parcel environment as recommended by United Parcel Service. The three types of bottles were:

1. 33 - 400 finish, Round, gallon size, HDPE, natural, non-fluorinated, 110 gram bottle weight (Figure 1).
2. 38 - 400 finish, Round, gallon size, HDPE, natural, non-fluorinated, 110 gram bottle weight (Figure 2).
3. 63 - 400 finish, F-style jug, gallon size, HDPE, natural, non-fluorinated, 125 gram bottle weight (Figure 3).

Both the round bottles were provided by Step Products Inc., Broadway, IL. The F-style bottle was supplied by Berlin Packaging Inc., Bellwood, IL.

2.2 Closure Types:

Three different types of closures were used on each of the bottle types described above. These were manufactured by Phoenix Closures, Naperville, IL.

1. Sure-Seal 200 series, a foamed LDPE core with top and bottom layers of 2 mil thick LDPE.
2. Foil-Seal 3/19, a heat induction foil lined closure.

3. Tekni-Seal closure liner, a pressure sensitive adhesive lined closure.

2.3 Package Configuration:

The three types of bottles were filled with 1 gallon (3.78 liters) of water. The bottles were then capped with the appropriate closures using the application torques which were recommended by the manufacturer. Careful closing techniques are used so that excessive torques are not applied that may damage the closure or bottle finish. The application torques were measured on all bottles. Six bottles of the same type with the same closure type were packaged in a regular shipping container that was recommended by the bottle manufacturer.

The 33-400 and the 38-400 round bottles, were packaged in single wall corrugated boxes with an ECT (Edge Crush Test) of 44 lb/in. as shown in Figures 4 and 5. The 63-400 F style bottles were packaged in single wall corrugated boxes, having an ECT of 32 lb/in. as shown in Figure 6. In all the above boxes, single wall corrugated dividers were used to enhance the structural performance of the box. A total of five boxes were made containing each of the three types of bottles and three types of closures. The application torques for all the closures and bottles were measured. The bottles that had the induction seal had to be re-tightened since they lost the application torque during the induction process. A total of 30 bottles for each type of bottle and closure were tested.

Two boxes containing a particular bottle and closure type were

Figure 1. 33-400 Round Gallon Bottle

Figure 2. 38-400 Round Gallon Bottle

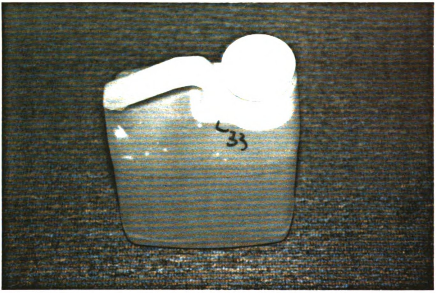

Figure 3. 63-400 F-Style Gallon Bottle

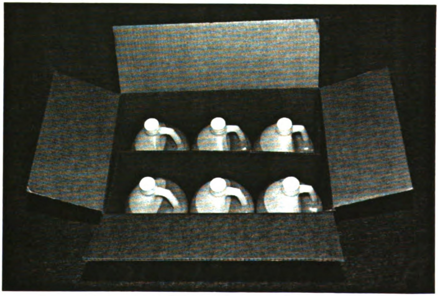

Figure 4. RSC for Six 33-400 Bottles

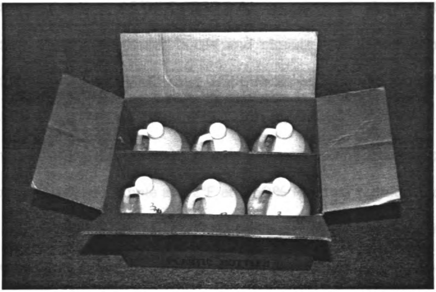

Figure 5. RSC for Six 38-400 Bottles

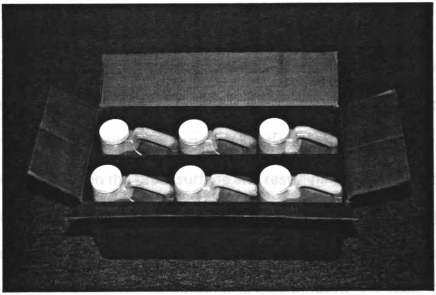

Figure 6. RSC for Six 63-400 Bottles

conditioned at normal conditions of 72°F and 50% Relative Humidity for 72 hours in accordance to ASTM D4332 prior to any tests. A second set of two boxes containing a particular bottle and closure type were conditioned at elevated conditions of 100°F and 85% Relative Humidity for 72 hours in accordance to ASTM D4332 prior to any tests.

In addition, a fifth box was containing a particular bottle and closure type was kept as a control sample at 72°F and 50% Relative Humidity for 72 hours in accordance with ASTM D4332. This box was not subjected to any tests but the removal torques for the bottles were measured after 72 hours. This data was collected to see the effect of creep in the plastic bottles and closures and determining the percent reduction of torque.

2.4 Package Vibration Test:

The RSC boxes containing the filled bottles after conditioning were subjected to a vibration test. The boxes each weighed approximately 53.5 lb. A Lansmont electro-hydraulic vertical vibration test machine was used. The boxes were placed on the table surface and restraining devices used to prevent the boxes from travelling sideways. The packages were subjected to a resonance scan from 3 to 100 Hz. The packages showed resonance at 5.8 Hz. They were then subjected to an input acceleration of 1.1 peak G to initiate repetitive bouncing. The duration of the vibration test was 45 minutes. These packages were then subjected to a drop test as described in 2.5.

2.5 Package Drop Test:

The boxes containing the bottles were subjected to a drop test in accordance with ISTA Project 1 A. A drop height of 18 inches was used based on the package weight of 53.5 lb. The box surfaces were identified as follows:

- top as one
- right side as two
- bottom as three
- left side as four
- near end as five
- far end as six

The boxes were subjected to ten drops using the following sequence:

- the 2-3-5 corner
- the shortest edge radiating from that corner
- the next longest edge radiating from that corner
- the longest edge radiating from that corner
- flat on one of the smallest faces
- flat on the opposite small face
- flat on one of the medium faces
- flat on the opposite medium face
- flat on one of the largest faces
- flat on the opposite large face

A Lansmont Precision Drop Tester was used to perform all the drops. In case of a bottle/closure failure during the drop test sequence, additional drops were not performed. However, removal torques were measured on the remaining bottles that were still intact.

All packages were then inspected for leakers. In addition the removal torque was measured on all bottles subjected to the vibration and drop tests and the control samples. The removal torques were measured using a Secure Pak Electronic Torque Tester. The same Torque Tester was used to measure both the application and removal torques on all bottles to avoid variation.

The data collected is discussed in the next chapter.

3.0 DATA AND RESULTS

The application and removal torques were determined for all bottles before and after performing the shipping tests. Table 1 lists these values for the 33-400 round bottles when subjected to shipping tests after conditioning at standard conditions of 72°F and 50% Relative Humidity for 72 hours. Two package samples were tested. The bottles were numbered 1 through 6, and identified "C" for corner and "M" for the middle location in the box. The torques were measured in in-lb. Table 2 and 3 lists these values for the 38-400 round bottles and 63-400 F-style bottles, when subjected to shipping tests after conditioning at standard conditions of 72°F and 50% Relative Humidity for 72 hours. Two package samples were tested for both these types of bottles.

Table 4 lists the torque values for the 33-400 round bottles when subjected to shipping tests after conditioning at accelerated conditions of 100°F and 85% Relative Humidity for 72 hours. Two package samples were tested. Table 5 and 6 lists these values for the 38-400 round bottles and 63-400 F-style, when subjected to shipping tests after conditioning at the accelerated conditions.

One package of each type of the three bottles were kept as a control sample for 72 hours at 72°F and 50% Relative Humidity. The application and removal torques were measured. These packages were not subjected to any shipping tests. Table 7 lists the torque values for the 33-400 round bottles. Table 8 and 9 lists the values for the 38-400 round bottles and 63-400 F-style bottles.

All the data for the torque measurements and number of leaks is summarized in Tables 10 and 11. Table 10 describes the average and standard deviation of the application and removal torques for all bottles tested. The bottles that had leaks resulted in zero removal torque values. Table 11 lists all the leaking bottles found after the various shipping tests.

The data from Table 11 shows that most of the leaks were found in the Sure-Seal type of closure. Also the 63-400 F-style bottle had the most number of leaks. This is probably attributed to the larger size finish opening. There is a larger variation in the finish dimension due to the larger size and also it has less material in the finish area as compared to the smaller finish bottles.

The 33-400 bottle showed the least number of leaks among all bottles tested. The bottles that were subjected to conditioning at higher temperature and humidity conditions showed a larger number of leaks. Also there was a greater reduction in removal torque values after they were subjected to shipping tests as compared to bottles conditioned at normal conditions.

Since each value of removal torque measured on a given bottle is dependent on the application torque for that bottle, it is important to determine the percent reduction of torque in all bottles tested based on the data in Tables 1 to 9.

TABLE 1: Application and Removal Torque for 33-400 Round Bottle (Normal Conditions)

Package Sample	Bottle Number and Location	Closure Type					
		Tekni-Seal		Sure-Seal		Foil-Seal	
		Torque (in-lb)		Torque (in-lb)		Torque (in-lb)	
		APP	REM	APP	REM	APP	REM
1	C1	20.5	6.2	16.9	3.6	22.4	8.1
	C2	20.0	5.7	21.5	4.2	21.0	7.6
	M3	19.2	5.2	16.5	Leak	20.5	7.0
	M4	21.1	4.9	19.9	4.4	19.5	7.1
	C5	19.0	3.2	18.1	2.5	22.1	8.0
	C6	20.6	5.0	18.6	4.2	23.2	7.2
2	C1	19.4	7.4	22.1	4.4	20.0	7.4
	C2	20.4	5.5	17.7	5.6	22.4	8.4
	M3	18.8	4.9	17.2	4.2	22.8	9.6
	M4	21.2	5.4	19.1	5.1	23.9	6.9
	C5	19.6	5.0	18.9	4.8	23.2	9.2
	C6	20.0	4.9	19.8	3.7	20.4	8.0

TABLE 2: Application and Removal Torque for 38-400 Round Bottle (Normal Conditions)

Package Sample	Bottle Number and Location	Closure Type					
		Tekni-Seal		Sure-Seal		Foil-Seal	
		Torque (in-lb)		Torque (in-lb)		Torque (in-lb)	
		APP	REM	APP	REM	APP	REM
1	C1	24.5	8.1	23.6	Leak	22.7	10.6
	C2	25.1	8.0	26.0	Leak	24.5	10.1
	M3	24.2	4.0	24.8	10.0	27.6	12.2
	M4	22.0	8.6	24.8	7.5	25.4	10.4
	C5	25.6	10.4	24.2	7.9	23.6	Leak
	C6	27.0	13.5	23.5	Leak	27.2	13.0
2	C1	26.5	8.8	24.2	6.2	26.7	Leak
	C2	26.0	9.5	23.5	6.2	29.0	12.7
	M3	26.6	8.5	23.1	7.3	25.1	Leak
	M4	27.0	10.9	24.1	10.4	25.6	10.0
	C5	27.8	8.3	26.1	7.8	29.0	11.8
	C6	29.8	8.4	22.4	6.7	27.1	Leak

TABLE 3: Application and Removal Torque for 63-400 F-Style Bottle (Normal Conditions)

Package Sample	Bottle Number and Location	Closure Type					
		Tekni-Seal		Sure-Seal		Foil-Seal	
		Torque (in-lb)		Torque (in-lb)		Torque (in-lb)	
		APP	REM	APP	REM	APP	REM
1	C1	40.4	20.4	41.5	16.1	42.4	Leak
	C2	39.5	25.2	40.5	17.7	43.1	Leak
	M3	40.7	21.5	43.4	Leak	41.3	27.6
	M4	45.0	13.3	40.9	19.0	42.1	26.0
	C5	45.2	Leak	43.2	13.9	39.8	28.3
	C6	48.7	21.9	42.3	14.1	41.2	36.7
2	C1	48.0	22.5	44.4	14.2	45.0	17.0
	C2	45.6	Leak	43.6	19.0	41.5	17.5
	M3	45.8	29.7	44.2	13.0	41.7	11.0
	M4	45.7	24.3	44.3	17.1	41.7	Leak
	C5	42.4	20.1	44.3	16.6	42.0	Leak
	C6	44.0	19.1	42.4	Leak	44.7	Leak

TABLE 4: Application and Removal Torque for 33-400 Round Bottle (Elevated Conditions)

Package Sample	Bottle Number and Location	Closure Type					
		Tekni-Seal		Sure-Seal		Foil-Seal	
		Torque (in-lb)		Torque (in-lb)		Torque (in-lb)	
		APP	REM	APP	REM	APP	REM
1	C1	20.0	4.6	19.3	1.9	18.9	7.7
	C2	20.3	4.0	19.1	2.6	20.8	6.4
	M3	19.9	3.1	21.1	2.5	20.0	6.8
	M4	18.6	3.8	19.8	2.3	21.4	4.9
	C5	20.7	5.1	18.4	3.7	20.1	5.4
	C6	17.8	3.8	21.5	3.4	20.9	8.3
2	C1	17.4	3.3	20.1	4.0	19.3	5.9
	C2	18.7	3.7	21.0	3.8	21.9	6.9
	M3	19.9	3.6	18.7	3.4	20.8	7.1
	M4	19.1	4.4	20.3	2.2	23.0	8.0
	C5	20.5	3.8	22.1	Leak	23.7	7.7
	C6	22.3	3.0	21.4	2.6	22.4	6.6

TABLE 5: Application and Removal Torque for 38-400 Round Bottle (Elevated Conditions)

Package Sample	Bottle Number and Location	Closure Type					
		Tekni-Seal		Sure-Seal		Foil-Seal	
		Torque (in-lb)		Torque (in-lb)		Torque (in-lb)	
		APP	REM	APP	REM	APP	REM
1	C1	20.3	2.5	28.2	Leak	23.1	Leak
	C2	20.5	2.0	26.9	5.5	23.5	Leak
	M3	21.4	2.9	28.3	6.3	23.2	8.1
	M4	19.6	2.2	25.3	4.8	25.6	9.9
	C5	20.6	Leak	25.1	5.3	23.6	Leak
	C6	21.6	3.0	27.4	5.3	24.0	Leak
2	C1	20.5	0.9	25.3	4.7	22.6	7.0
	C2	20.4	1.4	29.1	4.8	23.4	7.3
	M3	20.4	2.0	27.9	4.2	21.6	7.9
	M4	19.9	0.8	25.5	3.9	24.5	6.9
	C5	23.9	0.3	25.1	4.2	21.4	3.9
	C6	20.4	1.9	29.5	Leak	20.4	6.9

TABLE 6: Application and Removal Torque for 63-400 Round Bottle (Elevated Conditions)

Package Sample	Bottle Number and Location	Closure Type					
		Tekni-Seal		Sure-Seal		Foil-Seal	
		Torque (in-lb)		Torque (in-lb)		Torque (in-lb)	
		APP	REM	APP	REM	APP	REM
1	C1	39.4	5.9	45.6	10.2	41.4	Leak
	C2	46.4	8.6	39.7	Leak	42.9	17.1
	M3	38.2	6.4	41.5	7.3	39.7	20.6
	M4	39.5	7.0	49.2	11.0	40.6	21.8
	C5	43.2	9.2	44.3	10.9	42.2	17.1
	C6	40.0	Leak	39.7	Leak	37.2	20.9
2	C1	43.2	11.0	43.8	7.7	41.0	Leak
	C2	39.4	10.8	39.3	Leak	40.8	Leak
	M3	39.8	9.3	41.2	6.9	42.6	17.8
	M4	42.5	11.6	42.4	8.7	40.6	27.6
	C5	45.4	Leak	44.8	5.1	37.3	26.0
	C6	39.8	9.0	41.8	Leak	38.3	Leak

TABLE 7: Application and Removal Torque for 33-400 Round Bottle - Control Samples

Package Sample	Closure Type					
	Tekni-Seal		Sure-Seal		Foil-Seal	
	Torque (in-lb.)		Torque (in-lb.)		Torque (in-lb.)	
	App.	Rem.	App.	Rem.	App.	Rem.
33-400	19.4	7.6	18.6	5.8	21.0	8.1
	19.9	6.5	20.9	7.9	20.6	9.6
	19.4	7.9	21.3	7.1	23.4	14.1
	21.7	7.0	19.1	6.1	21.1	8.5
	20.6	7.1	19.2	5.7	22.4	10.1
	20.6	5.4	19.5	6.2	24.6	10.8

TABLE 8: Application and Removal Torque for 38-400 Round Bottle - Control Samples

Package Sample	Closure Type					
	Tekni-Seal		Sure-Seal		Foil-Seal	
	Torque (in-lb.)		Torque (in-lb.)		Torque (in-lb.)	
	App.	Rem.	App.	Rem.	App.	Rem.
38 - 400	25.3	9.1	28.2	8.2	21.7	9.6
	26.9	9.4	26.9	9.3	22.8	10.3
	26.6	10.6	28.3	7.9	28.1	13.2
	28.6	10.5	25.3	7.2	22.8	10.5
	22.1	10.4	25.1	6.6	23.6	11.3
	22.0	9.2	27.4	7.4	23.1	10.7

TABLE 9: Application and Removal Torque for 63-400 Round Bottle - Control Samples

Package Sample	Closure Type					
	Tekni-Seal		Sure-Seal		Foil-Seal	
	Torque (in-lb.)		Torque (in-lb.)		Torque (in-lb.)	
	App.	Rem.	App.	Rem.	App.	Rem.
63 - 400	41.7	19.1	45.2	18.6	36.6	26.2
	40.8	16.3	41.7	14.5	39.7	25.7
	44.4	16.8	41.0	13.8	41.8	28.0
	42.4	21.2	44.8	12.4	44.6	23.8
	39.0	15.2	41.2	15.1	40.5	25.8
	44.3	17.6	39.2	13.8	43.7	20.3

TABLE 10: Average and Standard Deviation of Application and Removal Torques Before and After Testing.

Condition	Bottle Type	Closure Type					
		Tekni-Seal		Sure-Seal		Foil-Seal	
		Torque (lb-in.)		Torque (lb-in.)		Torque (lb-in.)	
		APP	REM	APP	REM	APP	REM
Normal	33-400	20.0 (0.8)	5.3 (1.0)	18.9 (1.8)	3.9 (1.5)	21.8 (1.5)	7.9 (0.9)
	38-400	26.0 (2.0)	8.0 (2.2)	24.2 (1.1)	5.8 (3.8)	26.1 (2.0)	7.6 (5.7)
	63-400	44.3 (3.0)	18.1 (9.4)	43.0 (1.4)	13.4 (6.6)	42.2 (1.5)	13.7 (13.7)
Elevated	33-400	19.6 (1.4)	3.9 (0.6)	20.2 (1.2)	2.7 (1.1)	21.1 (1.5)	6.8 (1.1)
	38-400	20.8 (1.1)	1.7 (1.0)	27.0 (1.6)	4.1 (2.0)	23.1 (1.4)	4.8 (3.8)
	63-400	41.4 (2.6)	7.4 (3.9)	42.8 (2.9)	5.7 (4.5)	40.4 (1.9)	14.1 (10.9)
Control	33-400	20.3 (0.9)	6.9 (0.9)	19.7 (1.0)	6.5 (0.9)	22.2 (1.5)	10.2 (2.1)
	38-400	25.3 (2.7)	9.9 (0.7)	26.9 (1.4)	7.8 (0.9)	23.7 (2.2)	10.9 (1.2)
	63-400	42.1 (2.1)	17.7 (2.1)	42.2 (2.3)	14.7 (2.1)	41.2 (2.9)	25.0 (2.7)

TABLE 11: Number of Bottles With Leaks After Testing.

Conditions	Bottle Size	Closure Types No. of leaks		
		Tekni-Seal	Sure-Seal	Foil-Seal
Normal	33-400	0	1	0
	38-400	0	3	4
	63-400	2	2	5
Elevated	33-400	0	1	0
	38-400	1	2	4
	63-400	2	4	4
Control	33-400	0	0	0
	38-400	0	0	0
	63-400	0	0	0

The percent reduction for torque was determined for all bottles tested using the following equation:

$$\% R = \frac{\tau_A - \tau_R}{\tau_A} \times 100 \tag{3-1}$$

where,

	$\% R$	=	Percent Reduction in Torque, %.
	τ_A	=	Application Torque, lb-in.
	τ_R	=	Removal Torque, lb-in.

The Percent Reduction in Torque values calculated for the three different types of bottles are listed in Tables 12 to 20. Table 21 shows the average percent reduction in torque for all bottles tested. This data has been presented in histograms shown in Figure 7. In Figure 7 it is clear that in every case, there is a significant increase in the amount of torque reduction after the bottles are subjected to creep for 72 hours (control samples). These vary between 35 to 70 percent. The bottles subjected to the small parcel test simulation showed a further reduction. In addition the bottles that were conditioned at elevated conditions and tested showed the highest reduction in removal torques of as much as 90% in the 38-400 bottle with the Tekni-Seal closure. It can also be seen in Figure 7 that the Foil-Seal showed a higher reduction in torque on bottles that were subjected to the shipping tests as compared to the control samples. Similarly the bottles with the Tekni-Seal closure showed a higher reduction in bottles that were conditioned at the elevated conditions. In general the Foil-Seal closure had the lowest percent reduction in torque as compared to the Sure-Seal and Tekni-Seal closures.

Figure 8 shows the average percent reduction in torque due to creep (control samples) and the effect of shipping tests and elevated conditioning in the three bottle sizes. These values represent the average of all three types of closures within a finish size. The effect of creep during 72 hours of storage at standard conditions resulted in a 53.8% reduction in the 63-400 bottles, 61.5% in the 38-400, and 62.5% in the 33-400 bottles. The bottles subjected to shipping tests after 72 hours of conditioning at standard conditions showed a 64.8% in the 63-400 bottles, 70.9% in the 38-400, and 72.3% in the 33-400 bottles.

The bottles subjected to shipping tests after 72 hours of conditioning at elevated conditions showed the highest percent reduction in removal torques. These were measured to be 78.1% in the 63-400 bottles, 85.2% in the 38-400, and 79.1% in the 33-400 bottles.

TABLE 12: Percent Reduction of Torque in 33-400 Round Gallon Bottles after Testing and Conditioning at Normal Conditions.

Package Sample	Bottle Number and Location	Closure Type		
		Tekni-Seal	Sure-Seal	Foil-Seal
1	C1	69.75	78.69	63.84
	C2	71.50	80.46	63.81
	M3	72.91	100.00	65.85
	M4	76.77	77.88	63.59
	C5	83.15	86.18	63.80
	C6	75.72	77.42	68.96
2	C1	61.85	80.09	63.00
	C2	73.03	68.36	62.50
	M3	73.93	75.58	57.89
	M4	74.52	73.29	71.13
	C5	74.49	74.60	60.35
	C6	75.50	81.31	60.78
Average		73.59	79.49	63.79
Standard Deviation		4.94	7.85	3.60

TABLE 13: Percent Reduction of Torque in 38-400 Round Gallon Bottles after Testing and Conditioning at Normal Conditions.

Package Sample	Bottle Number and Location	Closure Type		
		Tekni-Seal	Sure-Seal	Foil-Seal
1	C1	66.94	100.00	53.30
	C2	68.13	100.00	58.78
	M3	83.47	59.68	55.80
	M4	60.91	69.76	59.06
	C5	59.38	67.36	100.00
	C6	50.00	100.00	52.21
2	C1	66.79	74.38	100.00
	C2	63.46	73.62	56.21
	M3	68.05	68.40	100.00
	M4	59.63	56.85	60.94
	C5	70.14	70.11	59.31
	C6	71.81	70.09	100.00
Average		65.73	75.85	71.30
Standard Deviation		8.19	15.41	21.34

TABLE 14: Percent Reduction of Torque in 63-400 F-Style Bottles after Testing and Conditioning at Normal Conditions.

Package Sample	Bottle Number and Location	Closure Type		
		Tekni-Seal	Sure-Seal	Foil-Seal
1	C1	49.50	61.20	100.00
	C2	36.20	56.30	100.00
	M3	47.17	100.00	33.17
	M4	70.44	53.55	38.24
	C5	100.00	67.82	28.89
	C6	55.03	66.67	10.92
2	C1	53.13	68.02	62.22
	C2	100.00	56.42	57.83
	M3	35.15	70.59	73.62
	M4	46.83	61.40	100.00
	C5	52.59	62.53	100.00
	C6	56.59	100.00	100.00
Average		58.55	68.71	67.08
Standard Deviation		21.44	15.52	33.25

TABLE 15: Percent Reduction of Torque in 33-400 Round Bottles after Testing and Conditioning at Elevated Conditions.

Package Sample	Bottle Number and Location	Closure Type		
		Tekni-Seal	Sure-Seal	Foil-Seal
1	C1	77.00	90.16	59.26
	C2	8.030	86.39	69.23
	M3	84.42	88.15	66.00
	M4	79.57	88.38	77.10
	C5	75.36	79.89	73.13
	C6	78.65	84.19	60.29
2	C1	81.03	80.10	69.43
	C2	80.21	81.90	68.49
	M3	81.91	81.82	65.87
	M4	76.96	89.16	65.22
	C5	81.46	100.00	67.51
	C6	86.55	87.85	70.54
Average		80.29	86.50	70.54
Standard Deviation		3.17	5.60	4.96

TABLE 16: Percent Reduction of Torque in 38-400 Round Bottles after Testing and Conditioning at Elevated Conditions.

Package Sample	Bottle Number and Location	Closure Type		
		Tekni-Seal	Sure-Seal	Foil-Seal
1	C1	87.70	100.00	100.00
	C2	90.20	79.55	100.00
	M3	86.40	77.74	65.09
	M4	88.80	81.03	61.33
	C5	100.00	78.88	100.00
	C6	86.10	80.66	100.00
2	C1	95.60	81.42	69.03
	C2	93.10	83.51	68.80
	M3	90.20	84.95	63.43
	M4	96.00	84.71	71.84
	C5	98.70	83.27	81.78
	C6	90.70	100.00	66.18
Average		92.00	84.64	78.96
Standard Deviation		4.69	7.51	16.35

TABLE 17: Percent Reduction of Torque in 63-400 F-Style Bottles after Testing and Conditioning at Elevated Conditions.

Package Sample	Bottle Number and Location	Closure Type		
		Tekni-Seal	Sure-Seal	Foil-Seal
1	C1	85.03	77.63	100.00
	C2	81.47	100.00	60.14
	M3	83.25	82.41	48.11
	M4	82.28	77.64	46.31
	C5	78.70	75.40	59.48
	C6	100.00	100.00	43.81
2	C1	74.54	82.42	100.00
	C2	72.59	100.00	100.00
	M3	76.63	83.25	58.22
	M4	72.71	79.48	32.02
	C5	100.00	88.62	30.29
	C6	77.39	100.00	100.00
Average		82.05	87.24	64.87
Standard Deviation		9.30	10.00	27.60

TABLE 18: Percent Reduction of Torque in 33-400 Round Style Bottles Due to Creep at Normal Conditioning.

Package Sample	Closure Type		
	Tekni-Seal	Sure-Seal	Foil-Seal
Control	60.82	68.8	61.43
	67.33	62.2	53.40
	59.27	66.7	39.74
	67.74	68.1	59.72
	65.53	70.3	54.91
	73.78	68.2	56.10
Average	65.75	67.4	54.22
Standard Deviation	5.23	2.8	7.69

TABLE 19: Percent Reduction of Torque in 38-400 Round Style Bottles Due to Creep at Normal Conditioning.

Package Sample	Closure Type		
	Tekni-Seal	Sure-Seal	Foil-Seal
Control	64.03	70.95	55.76
	65.06	65.42	54.82
	60.15	72.12	53.02
	63.29	71.53	53.95
	52.94	73.74	52.12
	58.18	73.00	53.68
Average	60.61	71.11	53.89
Standard Deviation	4.55	2.96	1.29

TABLE 20: Percent Reduction of Torque in 63-400 F-Style Bottles Due to Creep at Normal Conditioning.

Package Sample	Closure Type		
	Tekni-Seal	Sure-Seal	Foil-Seal
Control	54.20	58.84	28.42
	60.05	65.21	35.26
	62.15	66.32	33.01
	50.00	72.32	46.64
	61.03	63.32	36.31
	60.27	64.81	53.55
Average	57.95	65.15	38.86
Standard Deviation	4.78	4.38	9.37

TABLE 21: Average Percent Reduction of Torque in Bottles after Conditioning and Testing.

Condition	Bottle Type	Closure Type		
		Tekni-Seal	Sure-Seal	Foil-Seal
Normal	33-400	73.59	79.49	63.79
	38-400	65.73	75.85	71.30
	63-400	58.55	68.71	67.08
Elevated	33-400	80.29	86.50	70.54
	38-400	92.00	84.64	78.96
	63-400	82.05	87.24	64.87
Control	33-400	65.75	67.40	54.22
	38-400	60.61	70.11	53.89
	63-400	57.95	65.15	38.86

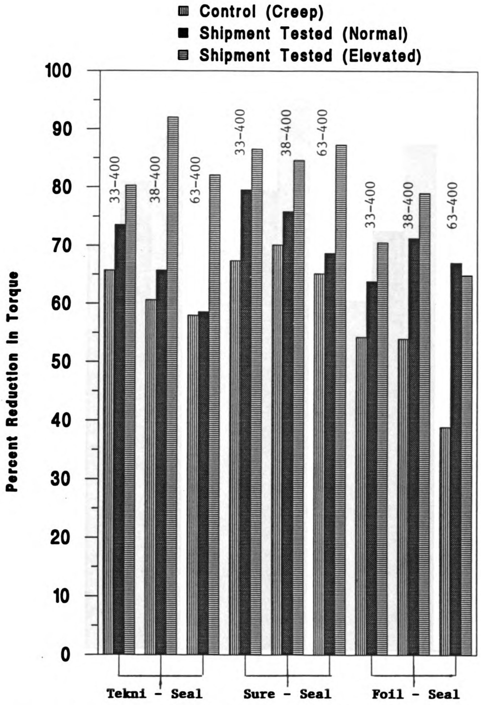

Figure 7: Percent Reduction in Torque in Gallon Size Bottles and Closure Type.

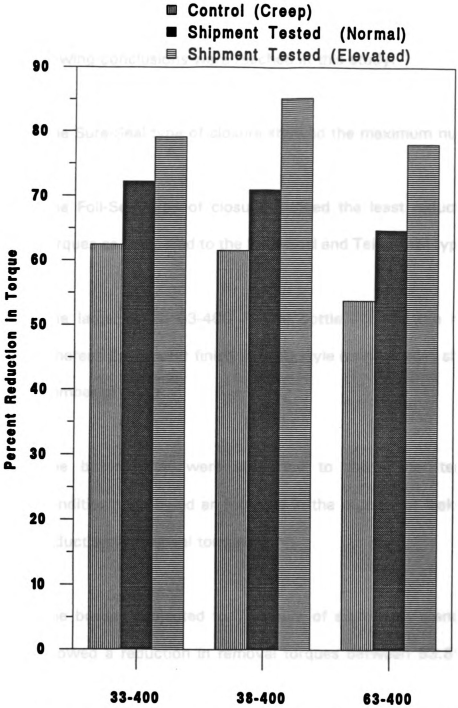

Figure 8: Average Percent Reduction in Torque in Gallon Size Bottles.

4.0 CONCLUSIONS

The following conclusions were reached in this study:

1. The Sure-Seal type of closure showed the maximum number of leaks.

2. The Foil-Seal type of closure showed the least reduction in removal torques as compared to the Sure-Seal and Tekni-Seal types of closures.

3. The larger finish 63-400 F-style bottle showed the maximum leaks whereas the smaller finish 33-400 style round bottles showed the least number of leaks.

4. The bottles that were subjected to the higher temperature and conditioning showed an increase in the number of leaks and a greater reduction in removal torques.

5. The bottles subjected to 72 hours of storage at standard conditions showed a reduction in removal torques between 53.8% and 62.5%. The bottles subjected to shipping tests after 72 hours of conditioning at standard conditions showed a reduction between 64.8% and 72.3%. The bottles subjected to shipping tests after 72 hours of conditioning at

elevated conditions showed the highest percent reduction in removal torques between 78.1% and 85.2%.

6. The location of bottles in the RSC had no significant effect on the number of leaks.

REFERENCES

Selected ASTM Standards in Packaging, Fourth Edition, ASTM, 1994

Hanlon, Joseph F., "Handbook of Package Engineering", Technomic Publishing Co., 1992.

McKearin, H. and K. Wilson, American Bottles and Flasks and their Ancestry, 1978.

Higdon, H., The Phoenix Flame, Phoenix Cap Company, Chicago, 1940.

Lief, A., A Close-up of Closures, Glass Container Manufacturers Institute, 1965.

Tang, T., "Closures, Liners and Seals", Wiley's Packaging Encyclopedia, John Wiley and Sons, 1984.

Phoenix Closures, Phoenix Cap Company, Chicago, 1984.

"Closures for Containers," Current Industrial Reports, U.S.Dept of Commerce, 1985.